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Papers

Enhanced Transmission, Wide Bandwidth RAR Nano-Textured Windows for Adsorption Resistant Gas Cells

by

James P. Nole,[*] Bruce D. MacLeod, Anthony D. Manni, and Stephen M. Consoles

ABSTRACT

Conventional thin-film anti-reflective coatings do not provide adequate reflection suppression and operational bandwidth for use in gas cells, where they exhibit a tendency to become fouled by deposits from chemical reactions within the cell. A new solution for AR treated cell windows is to replace the coatings with a nano-texture etched directly into the window material. The nano-texture consists of Randomly-distributed Anti-Reflective (RAR) nano-structures which provide unprecedentedly low levels of reflection loss across an extremely broad band of wavelengths and incident angles. No dissimilar materials are utilized in fabricating the nano-texture, thus there is no added surface absorption, resulting in an increased laser damage threshold for both pulsed and CW operation. Additionally, the nano-structures resist chemical adsorption and degradation, providing extended lifetimes of the cell windows while simultaneously providing unsurpassed optical performance.

Keywords: Nano-Texture, Anti-Reflection, AR, Motheye, Laser Damage, Organic Adsorption, Wavelength Reference Cells, Vapor Cells, Alkali-resistant

1.0 INTRODUCTION

Gas or vapor reference cells are used in applications where the wavelength of light needs to be accurately determined. The reference cell consists of a cylindrical type vessel that

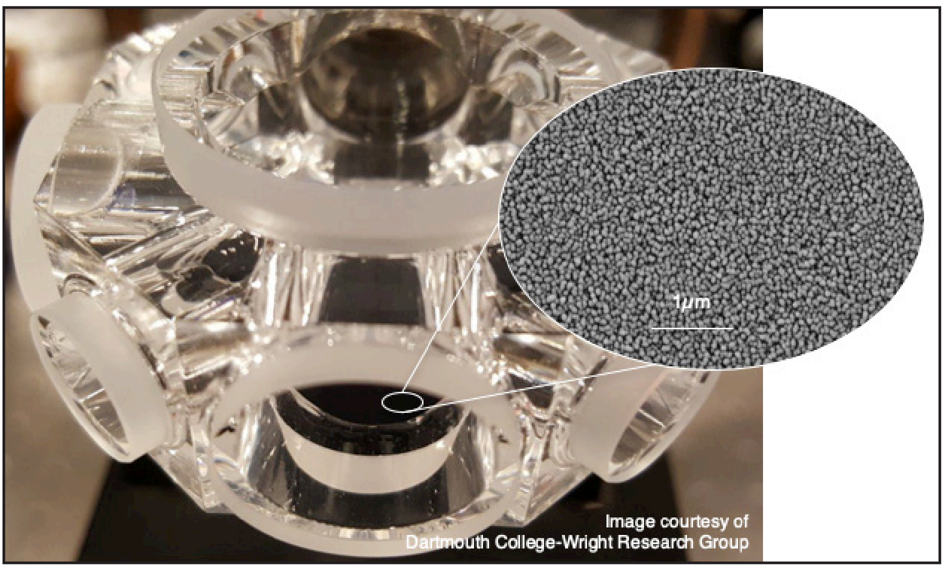

Figure 1. *Octagonal cell design manufactured by Precision Glass Blowing, Inc. for Wright Research Group Ultra-Cold Quantum Physics incorporating RAR nano-textured windows (Image Courtesy of Dartmouth College-Wright Research Group)*

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is filled with a high purity molecular compound or atomic element including Rubidium (Rb), Cesium (Cs), Iodine (I), Sodium (Na), Potassium (K), Thallium (Tl), or Indium (In). The cells are most typically assembled from Pyrex® or quartz with UV grade fused silica windows bonded or fused on each end. The fused silica entry and exit windows provide greater broadband transmission than Pyrex® or quartz and can be readily sourced with high level optical flatness for minimal wavefront distortion through the cell. Gas cells can be assembled using a variety of window sizes and designs including both planar and 'stepped' windows and can be built in standard tube and circular window designs to more complex configurations such as the octagonal cell design manufactured by Precision Glass Blowing for the Wright Research Group Ultra-Cold Quantum Physics group at Dartmouth College, New Hampshire (Figure 1).

2. BACKGROUND – NANO-TEXTURE ANTI-REFLECTION TECHNOLOGY

The conventional approach to suppressing reflections from optical windows is to deposit multiple thin layers of dielectric materials. The film stack is designed to effect destructive interference over a limited wavelength band and incident angular range. Many thin-film layers are needed to increase the spectral range over which reflections are suppressed. However, conventional thin-film coatings do not provide adequate reflection suppression or required bandwidth for many gas cell applications, and exhibit a tendency to become fouled by deposits resulting from chemical reactions within the cell. In many high-power laser applications, inherent absorption in the thin film coatings creates localized heating and distortion of the beam, and ultimately catastrophic failure of the optical component itself.

A new solution for AR treated cells is to replace the coatings with a nano-texture etched directly into the window material. The surface-relief structures provide ultra-low levels of reflection and extreme broad-band transmission, with none of the angle-dependent chromatic effects characteristic of multilayer thin film AR coatings. In addition, the RAR nano-texture eliminates surface absorption, exhibits the same chemical resistance as the substrate material, and has been shown to be naturally resistant to alkali and hydrocarbon adsorption. [1-6]

Antireflective nano-textured surfaces have been shown to be a superior alternative to thin-film AR coatings in many infrared and visible-band applications where low reflection, broad bandwidth, high angular acceptance, and environmental durability are critical. These nano-structures are fabricated directly into the surface of the window or optic material, imparting an optical function that minimizes reflections while maintaining the characteristics of the bulk material with respect to durability, thermal issues, and radiation resistance. The problems associated with thin-film coating adhesion, stress, and radiation hardness are removed by design as the thin films are eliminated. TelAztec fabricates three distinct types of surface relief microstructures, commonly known as **Motheye, SWS Effective Index, and Random Surface textures**. Each type of structure has unique characteristics and optical properties that can be tailored for specific materials and applications. TelAztec has given detailed descriptions of the three structure types in the literature, [2-5] a brief outline of which is given here:

2.1 MOTHEYE AR Structures: The Motheye structure is an array of surface depressions or pyramidal protrusions, such as those found on the eyes of nocturnal moths. [7-11] The tapered surface structure provides a gradual change of the refractive index for light propagating from air into the bulk optic material. [12-14] Reflection losses are reduced to a minimum for broad-band light incident over a wide angular range. A typical Motheye texture profile is depicted below left where the height h and the spacing Λ are indicated. A scanning electron microscope (SEM) image of Motheye structures fabricated in the surface of a CZT window are shown on the right below.

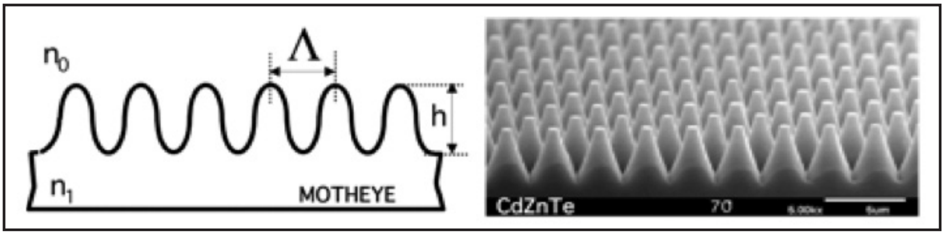


Figure 2. *Typical Motheye Profile (left); SEM of Motheye structure in CZT*

2.2 SWS Effective Index Structures: A Sub-Wavelength Structure, or "SWS" effective index texture is depicted in the profile diagram and SEM image below. An array of holes or posts provides an AR function that is equivalent to a single layer thin-film coating. The effective index is engineered to be the *ideal* index for the particular optic or window material, which is the square root of the material index of refraction. This is accomplished by tailoring the texture fill factor — the proportions of solid and open areas in the surface. Structure height *h* is then set to one quarter-wave optical thickness at the effective index. At one wavelength, reflections are completely eliminated, and over a narrow wavelength band, reflections are suppressed to very low levels. Dual and triple-band AR performance can be obtained with deeper structures set at multiples of the quarter-wave optical thickness. A scanning electron microscope (SEM) image of SWS binary structures fabricated in the surface of a CZT window, is shown on the right below.

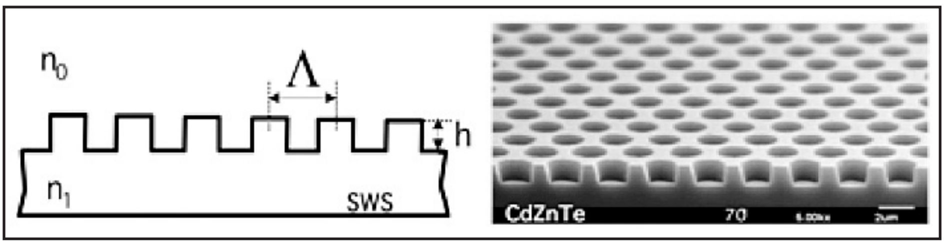


Figure 3. *SWS Type Binary Structure (left); SEM of SWS in CZT (right)*

2.3 Random Texture AR Microstructures: TelAztec has developed a simple fabrication process for AR textures that have a random distribution of sub-wavelength sized surface features. The very small and dense features, as shown in figure 4, provide AR properties that are extremely broadband. Advantages of the Random AR texture include the cost-driven benefit of eliminating the lithography step, and the ability to uniformly apply the texture to challenging topologies, such as stepped windows used in some gas cell designs as well as for fabrication on pre-existing patterned surfaces such as microlenses and MEMS. Random AR (RAR) nano-textures etched in fused silica are readily scaled to large areas and have been demonstrated in twenty-centimeter (20 cm) diameter high energy laser (HEL) exit apertures.

To achieve high performance AR with surface relief microstructures, optical phenomena such as diffraction and scattering must be avoided. This requires that the surface structures be fabricated with feature spacing (Λ in the figures above) smaller than the shortest wavelength of operation within the material for a given application. In addition, for

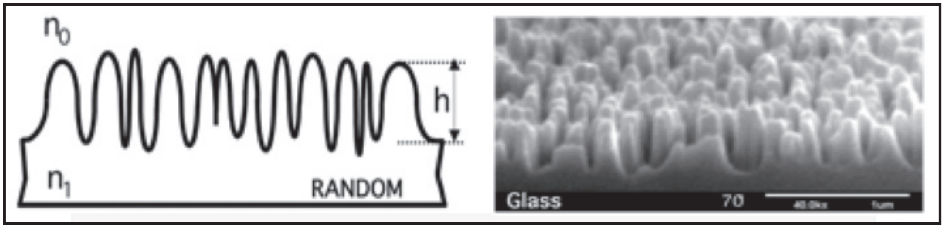
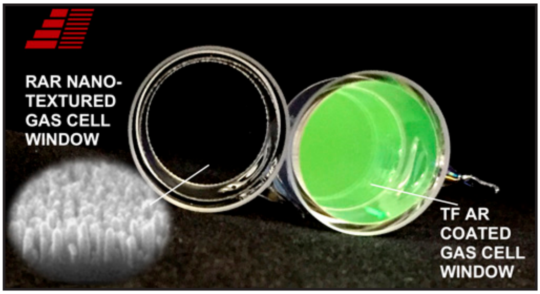


Figure 4. *Random AR nano-texture design (left); SEM image of Random AR in glass (right)*

Motheye and Random AR structures, the height and cross-sectional profile of the surface features must be sufficient to ensure a slowly varying effective index. In general, AR microstructures will exhibit similar characteristics as the bulk material with respect to mechanical durability, thermal issues, laser power handling capacity, and radiation resistance. The problems with thin-film AR coating adhesion, stress, off-axis performance, degradation, durability and lifetime are eliminated.



For applications with fused silica or glass windows and with operational bandwidth within the UV-SWIR range, RAR nano-structures have proven to be the optimal solution based on the durability, performance, and manufacturing cost.

3.0 RAR NANO-TEXTURE FABRICATION

RAR nano-structures are fabricated using a sequence of steps that are quite similar to the steps for depositing thin-film AR coatings, as illustrated in the process flow diagram of Figure 5. A batch of fused silica optics is first cleaned (1) and then placed into a carrier fixture (2) that is then loaded into the plasma etch tool vacuum chamber. An RAR etch recipe is then initiated under computer control (3). The etch recipe may include options such as a pre- or post-RAR argon or oxygen ion cleaning, and all etch parameters such as temperature, pressure, power, gas composition and gas flow rates are computer controlled during the run. Typical cycle times for fused silica optics discussed herein were about 20 minutes from load to unload (4), much faster than typical coating deposition cycles.

The plasma etch process for RAR nano-texture has an inherent "depth of process" ability that allows uniform etching over varied topology such as lenses and stepped windows. TelAztec has shipped over 15,000 nano-textured optics for insertion into both pulsed and

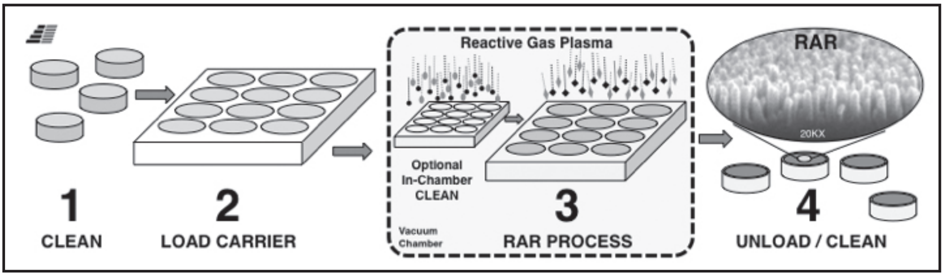


Figure 5. *Process flow diagram for the batch fabrication of the RAR texture*

CW laser systems as well as specialty products such as gas cells. Figure 6 on the right shows a nano-textured stepped window, where the stepped perimeter is left polished to allow for optimal sealing in a gas cell. There is minimal reflection at all angles within the textured central aperture. Recent advancements have been made by TelAztec customers in the bonding of nano-textured windows to gas cells by using fritted glass powder in an adhesive during the fusing process.

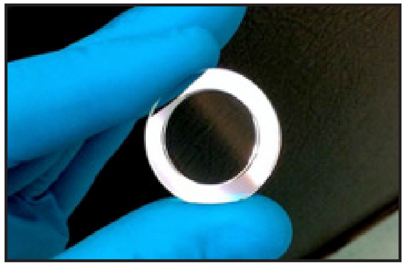


Figure 6. *RAR textured 'stepped' nano-textured window*

4.0 MEASURED PERFORMANCE OF RAR IN FUSED SILICA

RAR nano-textured fused silica optics provide exceptionally low broadband reflectance levels, and are commonly used for UV through NIR, and visible through IR applications. The gradual variation in index provides unprecedented off-axis performance with minimal change in reflectance from +/- 45° and beyond.

4.1 Reflection suppression and bandwidth: The Random Anti-reflective nano-texture can be optimized for ultimate reflection levels less than 0.05% at discrete customer specified laser lines, such as 266, 355, 532, 1064, and 1535 nm. Figure 7 shows RAR designs for commercial applications that include frequency doubling. Common options include RAR-S for applications requiring anti-reflection from 250 nm-550 nm; RAR-L for applications requiring anti-reflection from 500 nm-1100 m; RAR-L2 for applications requiring the reflection minimum at the 1064 nm laser wavelength; and RAR-VL for extreme broad-band applications from the visible band to 2000 nm.

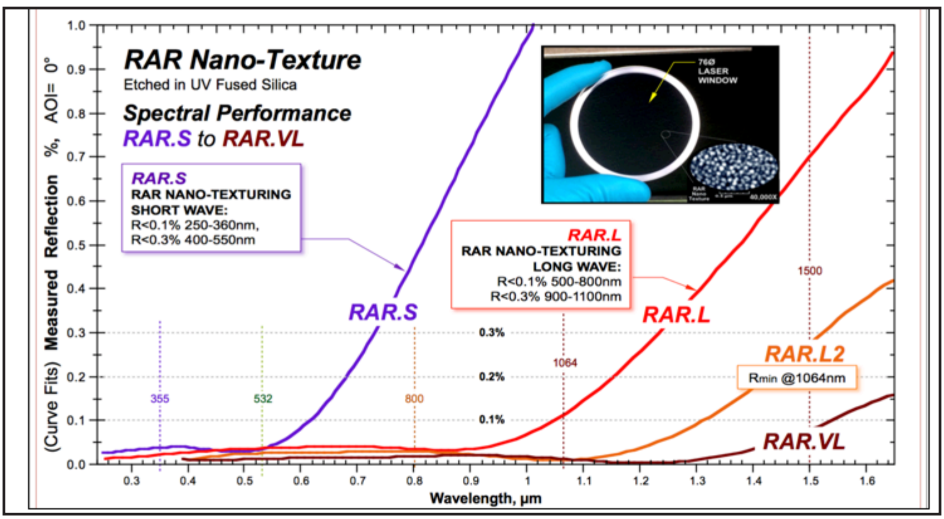


Figure 7. *Commercially available RAR Performance Options: RAR-S, RAR-L, RAR-L2, and RAR-VL*

4.2 Extreme Off-Axis Performance, Angle of Incidence (AOI): The RAR nano-texture is designed to perform as a graded index surface, providing a gradual change from air to the bulk substrate. The gradual change in index remains even at steep angle of incidences, allowing the surface to perform well out to 60° AOI and beyond. Figure 8 shows RAR reflection performance measured from 8° through 55° angle of incidence, across the extreme 350-1100 nm bandwidth. The reflection remains below 0.5% across the visible band out to

55°. Thin film coatings cannot perform to this level and exhibit a color shift to shorter, out of band wavelengths due to the inherent thin film properties of interference.

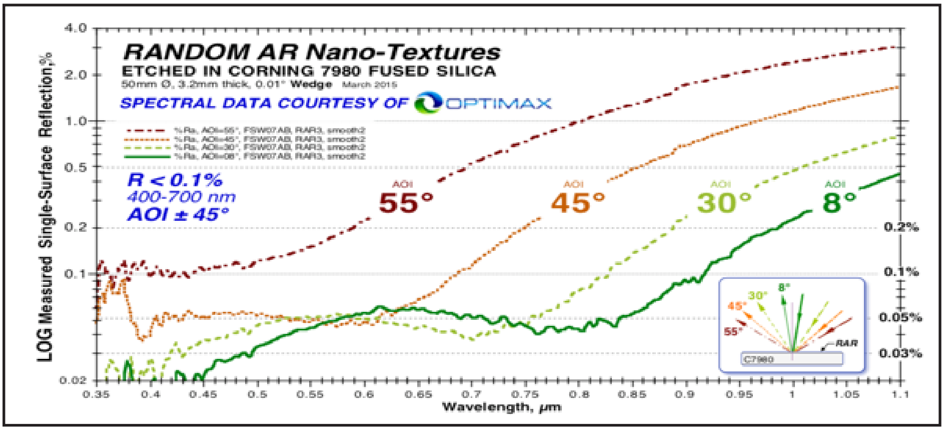


Figure 8. *Off-axis broadband performance of RAR nano-textures in fused silica out to 55° (Spectral Data courtesy of Optimax)*

5.0 SURFACE ABSORPTION OF RAR IN FUSED SILICA

Thermal effects related to surface absorption are the most common causes of beam quality degradation and optical component failure. Because the RAR nano-texture solution does not employ or deposit any dissimilar materials, there is no added absorption. To compare the surface absorption of nano-textures to low absorption thin-film anti-reflection coatings on fused silica, surface-discriminating absorption measurements have been made using the Photothermal Common-path Interferometry (PCI) method (Stanford Photothermal Solutions, Inc.). The results are seen in Figure 9, where absorption is measured through one surface at 1.5 mm, goes through the bulk and exits the back surface near 5.5 mm. Zero change in surface absorption is seen for the RAR nano-textured optics, as revealed by the scan data of both as-polished (gray line) and RAR nano-textured fused silica (green line). A large absorption spike is seen in the thin film coated sample (red line) on the coated surface, with a 3 part per million level – which is typical for thin-film AR coated optics.

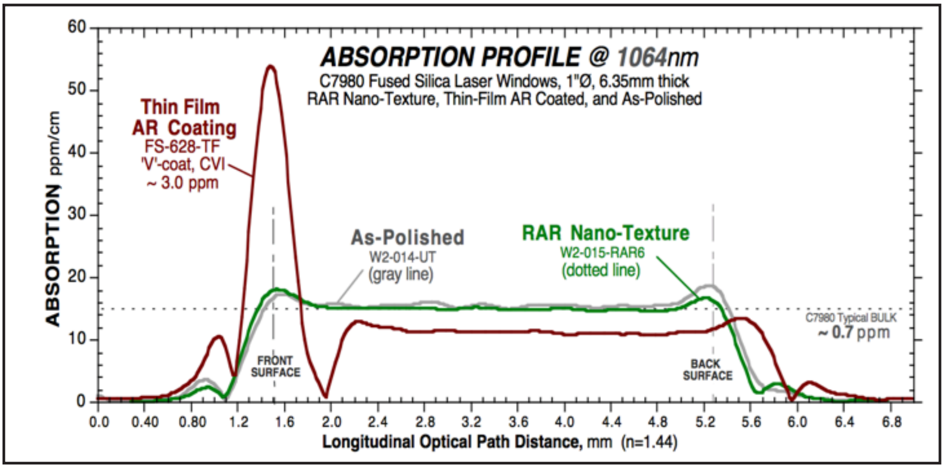


Figure 9. *Longitudinal CPI absorption scans through as-polished, RAR treated, and thin- film coated damage test samples*

A more problematic issue with coatings is the presence of large absorption spikes across the surface, which are attributed to coating defects. These absorption "hot spots" are considered to be precursors to failure for coatings as they exhibit extreme localized heating under high fluencies which leads to failure at unpredictable levels.

To obtain a greater understanding of surface absorption uniformity, the overlapped PCI measurement beams were transversely scanned across the surface of the substrates instead of through the bulk as in Figure 9. Figure 10 shows the surface absorption result of 9 mm long transverse scans across the surfaces of as-polished, RAR nano-textured, and thin-film AR coated samples. The background absorption level for the coating is constant across the surface at ~2.5 ppm, yet many significant absorption spikes are revealed. These elevated absorption spots can produce extreme temperature rise under high irradiance leading to a number of failure mechanisms. The untreated and RAR textured sample surface absorption levels are nearly constant across the surface, and the measured level between 0.5-0.7 ppm is related to bulk absorption rather than surface absorption.[6]

It should also be mentioned that the small surface absorption spikes observed on untreated and RAR nano-textured optics are typically negligible levels of contamination related to inadequate cleaning. This has been revealed in cleaning tests on RAR textures where these small absorption spikes are removed. Under high laser powers, residual contamination on the tips of the RAR nano-structure can be ejected or evaporated easily, whereas contamination sealed underneath a thin film coating is permanent.

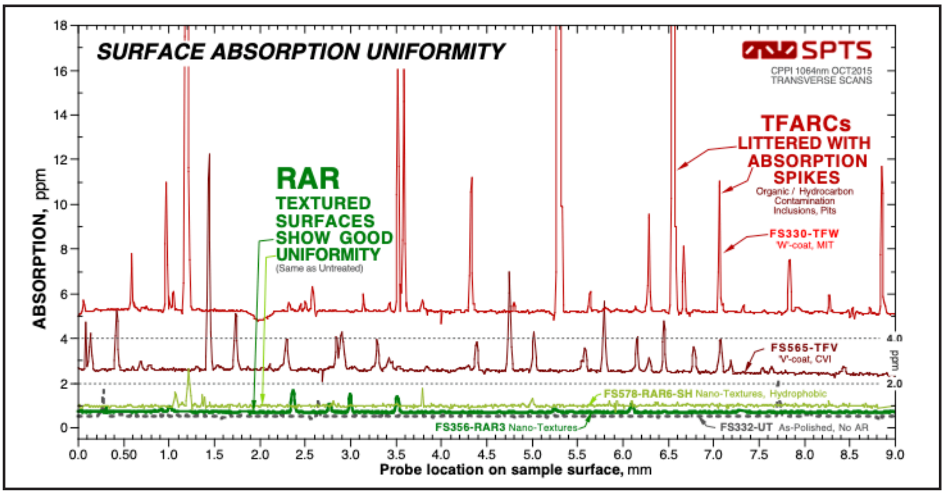


Figure 10. *Transverse CPI surface absorption scans through untreated (gray), RAR treated (green), and thin-film AR coated (maroon) fused silica samples*

6.0 CONTAMINATION RESISTANCE OF RAR IN FUSED SILICA

The materials that comprise thin film coatings often suffer from environmental durability issues, a particular issue for vapor cells. The chemical vapors employed by reference cells, Diode Pumped Alkali Lasers (DPAL), Spin Exchange Optical Pumping (SEOP) and other gas cells can attack film coatings during laser operation. [16,17] Chemical damage and carbon fouling of the gas cell windows can lead to catastrophic surface damage. Windows of the cell containing the alkali vapor typically become fouled with deposits or fogged by damage from chemical reactions with the gas, reactions that increase as the laser optical power is scaled up. One of the primary benefits of the RAR nano-texture solution for gas and vapor cell windows is the inherent contamination resistance of RAR. Since the RAR nano-texture is a textured surface (non-flat) integrated directly into the surface of the window material, other materials are eliminated by design and the nano-texture retains that same chemical durability as the window itself. Due to the high aspect ratio of the needle-like AR nano-structures, the nano-texture has a certain degree of inherent hydrophobicity without any additional processing. Extreme levels of hydrophobicity are attainable with further treatment after etching the RAR nano-texture.

In controlled vacuum outgas testing, RAR textures in fused silica windows were subjected to capillary condensation tests to evaluate the resistance of the RAR texture to the adsorption of organic compounds. It was found that for a one-day exposure time to a combination of organic contaminants, the RAR textured fused silica surfaces adsorbed up to 200 times less than high end coatings.[6] Additionally, transmission of the RAR textured windows remained unchanged after multiple exposure cycles. The RAR textured windows also allowed for an aggressive sulfuric acid and hydrogen peroxide chemical cleaning cycle to be performed after each exposure with no adverse effects on the RAR performance, further providing evidence of the long-term durability and cleanliness of RAR nano-textured surfaces. In other testing, RAR treated windows showed no physical damage from alkali exposure in diode pumped alkali (DPAL) and testing operating conditions, and were further shown to be easily cleaned and to retain all pre-exposure characteristics.

7.0 LASER DAMAGE THRESHOLD OF RAR NANO-TEXTURES

Nano-structured optical surfaces have been proven to exhibit higher performance and improved long term survivability than thin-film coatings for an increasing number of materials used within high energy laser (HEL) systems. Thin-film coated laser optics exhibit surface absorption related heating issues, which lead to reduced beam quality and eventual component failure in both CW and pulsed laser systems.

For evaluation of RAR nano-textured fused silica optics in pulsed laser applications, standardized laser induced damage threshold (LiDT) measurements were completed using commercial testing services. The damage thresholds were determined for untreated, RAR treated, and thin-film coated optics at important laser wavelengths from 355 nm through 1538 nm, as shown below in Figure 11. The LiDT of RAR nano-textured sam-

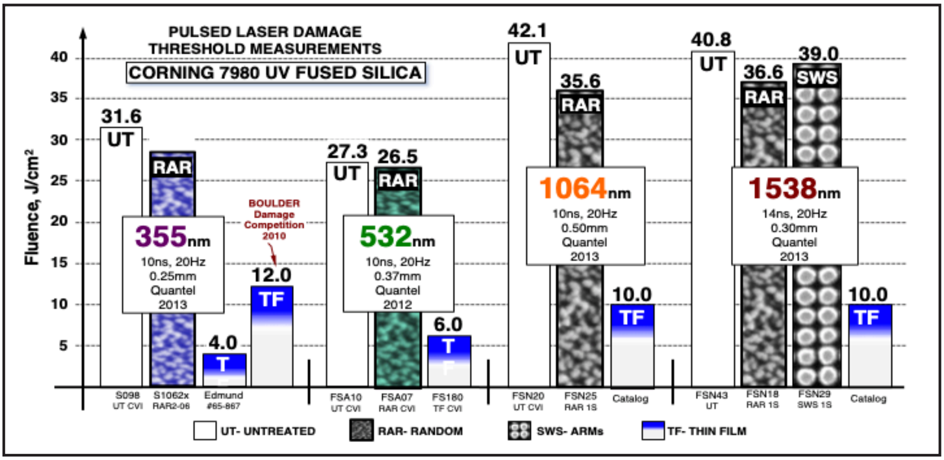


Figure 11. *Pulsed Laser Induced Damage Threshold Comparison: Untreated, RAR Nano-textured, and Thin-Film Coatings at important laser wavelengths*

ples was typically found to be comparable to that of uncoated samples, and 4-8 times greater than the level specified by prominent TFARC providers.

To demonstrate the power handling of nano-textured optics in CW laser applications, a series of laser damage tests at 1070 nm demonstrated that RAR textured fused silica optics could not be damaged up to the maximum available power density of 15.5 MW/cm^2.[15] Testing was completed at the Pennsylvania State University's Applied Research Laboratories Electro-Optics Center (PSEOC) with an IPG Photonics Model YLS17000 Fiber laser capable of 17kW of CW output power at 1070 nm. Thermal imaging at maximum irradiance showed minimal laser induced heating of RAR textured samples, similar to results seen with polished fused silica. In contrast to these results, the thin-film coated samples show widely variable damage thresholds as low as 1 MW/cm^2, and thermal measurements revealed occasional spikes in temperature up to hundreds of degrees Celsius prior to failure, as shown in Figure 12. The CW damage results are attributed to levels of surface absorption for each AR treatment, as described above. Photothermal Common-path Interferometry (PCI) measurements showed no evidence of surface absorption for RAR textured fused silica windows, consistent with the absence of deposited materials. The thin-film AR coatings exhibited surface absorption levels of 2-5 ppm, as well as the presence of large absorption spikes across the surface, which are attributed to coating defects. These absorption "hot spots" are considered to be precursors to failure for coatings as they exhibit extreme localized heating under high fluencies, which can lead to failure at unpredictable levels.

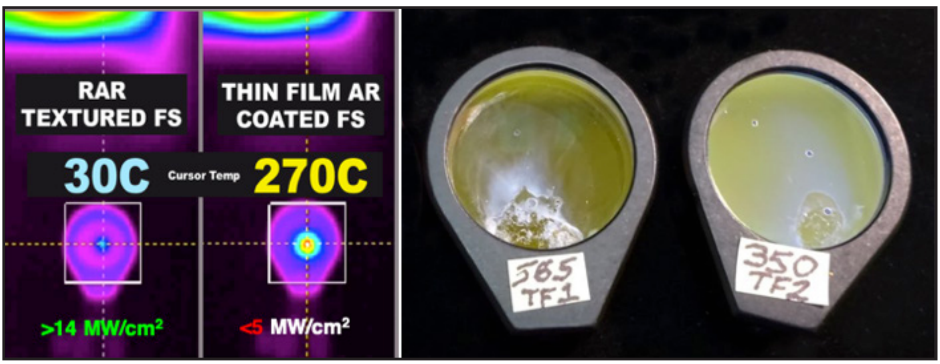


Figure 12. *Thermal images of 30sec CW laser exposures at 1064 nm for RAR nano-textured and thin-film coated fused silica windows (left). Image of CW laser-damaged thin-film coated optics (right).*

8.0 SUMMARY

Random AR nano-textures (RAR) can be generated in a variety of glass types and provide unsurpassed optical performance and increased lifetimes versus traditional thin-film anti-reflection coatings. For gas and vapor cells, RAR nano-textures can be generated in a variety of size and shape windows and can be integrated cost effectively into current cell designs. Optically, RAR nano-textures can achieve performance down to 0.01% reflectance and can be designed to perform over unprecedented wavelength spectrums from the UV through the NIR. Environmentally, RAR nano-textures in fused silica have been shown to be hydrocarbon resistant as well as resistant to the chemical damage and carbon fouling due to adsorption typically seen with AR thin-film coated cell windows. Finally, because there is no added absorption and thus no heating of the surface, RAR nano-textures have demonstrated a 4-8X increase in pulsed laser induced damage threshold (LiDT) levels over thin-film coatings and in several rounds of CW LiDT at 1070 nm could not be damaged up to the maximum power density of those tests of 15.5 MW/cm2, a level 3-9x greater than thin-film anti-reflection coatings.

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The authors gratefully acknowledge work done integrating RAR anti-reflection nano-textured glass windows by Jay Meikrantz and Ron Bihler at Precision Glass Blowing (PGB) of Englewood, CO and Kevin Wright at the Wright Research Group Ultra-Cold Quantum Physics at Dartmouth College in New Hampshire for the exceptional images of octagonal cells manufactured by PGB and utilized in the Quantum Physics laboratory instruments.

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From the Alchemical to the Mundane

by

Jim Hodgson*

ABSTRACT

If your glassware is located a one-day jeep ride on a rugged trail, a one-day hike on foot beyond that, and there are no scientific glassblowers nearby, it is well to consider the most robustly designed glassware possible. Glass is the perfect material for distilling and separating essential oils, but complex apparatus may not be the best choice when repairs or replacements are not readily available. The distillation of Vetiver oil in Papua, Indonesia, required some reexamination of what was readily available to come up with a solution both robust and practical.

BEGINNINGS AND ENDINGS

Most scientific glassblowers enjoy working on apparatus that is interesting and challenging. In the two photos below, the one on the left is the job we would all enjoy, but sometimes the best solution is not the one that is the most fun. In this case, the design process was most of the fun. That, and getting to be part of something that would have a positive impact on people's lives (Photos 1 & 2).

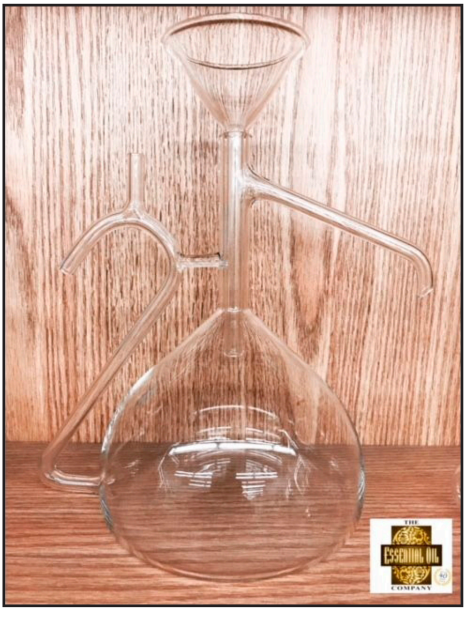

Photo 1. *The Alchemical*

Photo 2. *The Mundane*

LOCATION, LOCATION, LOCATION

The work began with a call from a former Kansas State engineering student. He requested help with a water/oil separator for essential oil distillation. The separator would be used by the Meyah indigenous people of Papua, Indonesia. Access to the area was a one-day jeep ride if the roads were passable and a one-day hike on foot. The essential oil was distilled

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from Vetiver root. Vetiver root serves two main purposes. The deep roots of the Vetiver plant work well to mitigate erosion on slopes up to 70 degrees in an area with mountains over 16,000 feet tall and tropical rainfall. The essential oil which could be distilled from these roots is used in 90% of perfumes, contains over 150 aromatic compounds, and is a potential good source of income for the Meyah (Photos 3 & 4).

Photo 3. *The Vetiver plant*

Photo 4. *A bundle of Vetiver roots*

PORTUGUESE ALEMBIC STILL AND SEPARATOR

Photo 5. *Portuguese Alembic copper still and separator*

"Alembic" just reeks of mystery and the alchemical, does it not? This alembic copper pot still utilizes steam distillation to distill a beautiful emerald green oil of high quality. It takes 55 pounds of Vetiver root to yield only 40 ml of oil (Photo 5).

The original design of the oil/water separator was prone to breakage due to the long lever arms and small diameter tubing. The modified design, while it utilized replaceable component parts suffered from the same problems and the Teflon™ grommets were not a realizable solution from a glassblowing perspective. Not to mention, there were no local glassblowers (Photos 6 & 7)!

A ROBUST ALTERNATIVE

The relative fragility of glass necessitated a change to a stainless steel separator with a sanitary sight glass at the top. The sight glass utilized stainless steel with Tri Clover compatible fittings and a borosilicate cylinder to

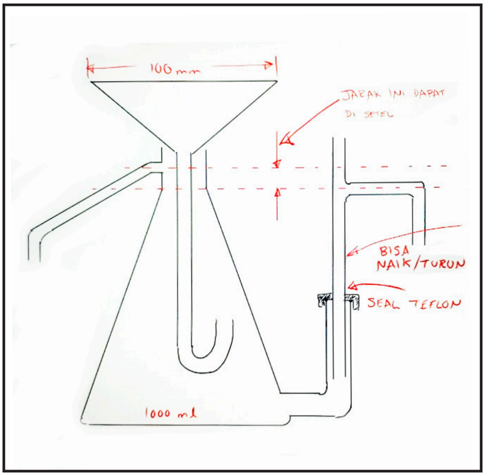


Photo 6. *Original separator*

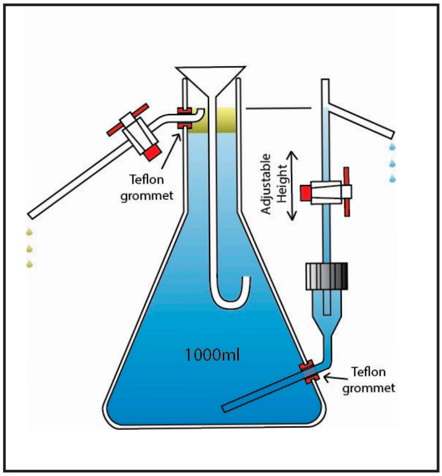


Photo 7. *Modified separator*

visually monitor and adjust the separation process. This stainless hardware is robust, easily cleaned, relatively inexpensive, and readily available. The sight glass does require the addition of a side port to take off the distilled Vetiver oil. Although this is hardly romantic, there are still several factors to be considered in both design and fabrication.

The example picture shows a short Teflon™ stopcock sealed to the cylinder. This still has too much chance of breakage and the stopcock is not really necessary. But even a simple side port will present challenges in sealing to a very short (4”), very heavy walled (5 mm) glass cylinder. The job would be simplified if the sight glass tubing was readily available, but nothing of that diameter and wall thickness could be found despite an exhaustive search of suppliers (Photos 8 & 9).

SIDE PORT AND TUBING SELECTION

The most robust solution for the side port was to simply seal on a short hose barb which would accept a short length of tubing to collect the Vetiver oil as it was separated. A hose barb which would fit 3/8” i.d. tubing was chosen as the most practical, strongest option. The sight glass and extra borosilicate cylinders were ordered.

Photo 8. *Sight Glass with Tri Clover fittings*

Photo 9. *Example of a Side Port on Sight Glass*

Essential oils can be aggressive solvents so Ultra-Chemical-Resistant Versilon PVC Tubing was chosen. It is clear, flexible, works with hose barbs and is lined with FEP for good chemical resistance. It is readily available in various sizes from McMaster-Carr.

It is good practice to check the size of tubing which will be used to make sure it will be a snug fit on the side port. It is especially important when you only have one chance to get it right. Always ask your customer to provide a sample of the tubing if available. I keep samples of many different types of tubing to inform myself and show customers. They are well labeled with part numbers and vendors so they will be easy to order or locate. I ordered the 3/8" i.d. tubing sample to check the fit and add to my collection of tubing (Photo 10).

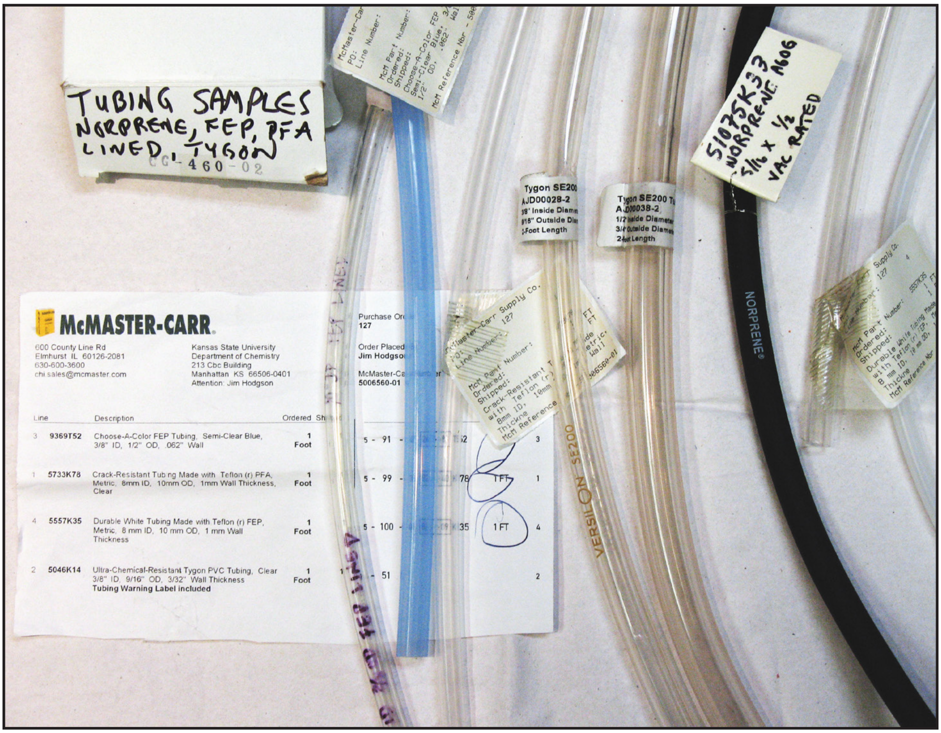


Photo 10. *Various tubing samples with labels*

The tubing was checked for fit on each hose barb I planned to use as the side ports. This is also important as there can be variations in stated dimensions as well as differences between individual hose barbs.

THE COLD SEAL SOLUTION

A side seal is usually a simple thing, but not so simple when it is to a 4" length of very heavy wall tubing. Any plugs used to block the ends and provide a way to blow are likely to burn and even holding the short length while sealing is a problem. Due to the thickness of the wall, the tubing must be kept warm and flame annealed extensively just to survive before oven annealing to remove stress.

This is the perfect place to utilize a cold seal. I learned the technique from Allan Brown and Bill Wassemiller. The technique is simple and fairly reliable.

1. Warm the end of the working piece of glass.
2. Get the end of the glass you will use as a handle red hot, let it cool slightly but not lose all color, and stick the two together gently.

The warm end of the important working part will not distort using this technique and as long as you do not apply physical force to the temporary cold seal or get it near a flame, you will be able to do the necessary work. When your work is complete, just apply a small sharp flame to the cold seal and it will fall apart. (*Caveat*: This seal is only temporary and may fall apart at the worst possible time. It is well to practice the technique and become comfortable with it to use it successfully, but it is very useful and well worth the practice (Photos 11 & 12.)

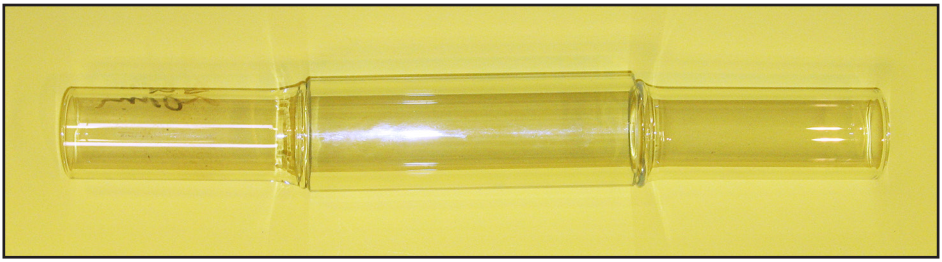


Photo 11. *Cold sealed handles*

Photo 12. *Finished sight glasses*

THE FINISHED SEPARATOR

The finished separator in stainless steel looks not unlike the alchemical glass separator and was fabricated by a local company with Kansas State ties also. Kansas State alumni help each other out! Of course the obligatory flame picture with the Kansas State engineer before heading to Papua, Indonesia (Photos 13 &14).

VETIVER OIL COLLECTION

The finished separator was placed in a water bath to help with temperature control and more efficient separation. The beautiful emerald green Vetiver oil can be seen in the sight glass as it flows out through FEP lined tubing (Photo 15).

Photo 13. *The end product*

Photo 14. *A satisfied customer*

CONCLUSION

The sight glass on top of the stainless steel separator was a long way from the original design and thoughts, but the journey to get there was very interesting and presented some glassblowing challenges.

There is satisfaction in solving a problem and helping others, and as my engineering friend said when he asked for a picture of me with the separator, "We like to keep pictures of the various folks around the world who have 'chipped in' to make these projects go…it encourages our hearts and those of the Meyah tribe as well."

ACKNOWLEDGEMENTS

I would like to express my appreciation to Kansas State University and the Department of Chemistry for their support of the scientific glassblowing facility and their continuing encouragement in my professional endeavors. And to those glassblowers who, by example and teaching, encourage a desire to do good work. Special thanks go to Kansas State alumni Michael Cochran and the Meyah indigenous people of the Arfak mountain area of Papua, Indonesia.

Photo 15. T*he separator in action*

Robots, Lasers, 3D-Print —
What is the Future of Scientific Glassblowing?

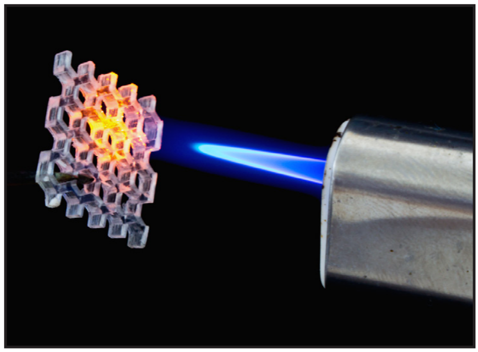
by

Klaus Paris*

ABSTRACT

Thirty five years ago, scientific glassblowing was still very much a traditional craft. Today, in 2019, scientific glassblowers are confronted with creating an entirely new scope of apparatus and using new and unique technologies in order to meet the demands of the ever changing world of science. This paper will give an overview of a few technologies that are possible today and invites everyone to bring their own creative vision for the future of scientific glassblowing.

Glassmaking has changed and improved countless times in the last 5000 years since its discovery. However, the main processes in glass production have remained. Scientific glassblowing is an ongoing process of improvement and facing new challenges as science requires steadily new solutions made with glass. Now we are in the midst of a new glassmaking technology based on laser, 3D-printing and robots. New challenges, additional to glassblowing skills, will be required.



3d printed quartzglass honeycomb

ROBOT-BASED AUTOMATION

What makes a successful robot application? Basically, an industrial robot can only move a fictitious working point (TCP = Tool Center Point) quickly, precisely and repeatably within a defined workspace. It is robots or tools guided by robots, the supporting sensors and sophisticated software, intelligent controls and the programming or configuration by qualified personnel that make them the most efficient and versatile machines for automation.

PICK AND PLACE OF HIGHLY SENSITIVE GLASS TUBES WITH FLEXIBLE COBOTS**

Hofmann Glastechnik GmbH wanted to make better use of their employees' potential and aimed to optimize its production processes.

This is why Hofmann Glastechnik started to automate repetitive, manual tasks. Now, the family business uses two collaborative robot arms from universal robots, automatically feeding highly sensitive glass tubes into a forming machine. As a result, the production process has been stabilized, the quality of the glass components has been improved significantly, and the employees have been effectively relieved from monotonous tasks. So the company was able to increase its production capacities in the specific application area by 50 percent and the original investment in the robots was paid off in around six months.

The two robot arms UR5 and UR10 work at several CNC glass lathes. They tend the ma-

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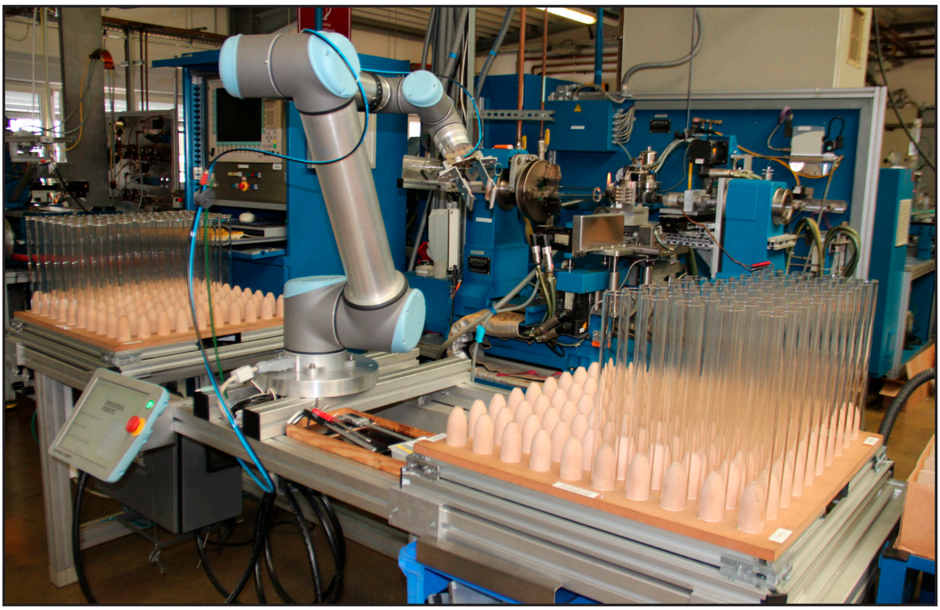
** Cooperating robots.

chine for up to 11 hours each day. If required, the company can flexibly implement the robots at different glass lathes. Therefore, the company has developed a facility which allows them to switch the UR robots between the different machines with no problems at all. The robots are immediately ready for operation after the set-up has been changed in this way. Björn Uthe, Department Manager Machine Park at Hofmann, is also convinced of the usability of Universal Robots. “Handling the UR robots is very intuitive. Once you have understood the procedures and functions involved, literally anyone can operate the robots.”[1] Thereby, robot arms also lighten the work load for the skilled workers at Hofmann. “The UR robots have made our work so much easier. We used to have to move backwards and forwards between the glass lathes, parallelly feeding them all day long. We could hardly keep up with the production. With the UR5 and UR10 working with us now, I can spend more time on setting up the glass processing machines,” explains Björn Uthe.[2]

www.universal-robots.com/ [2]

hofmann-glas.com/en/ [2]



Hofmann Glas robot

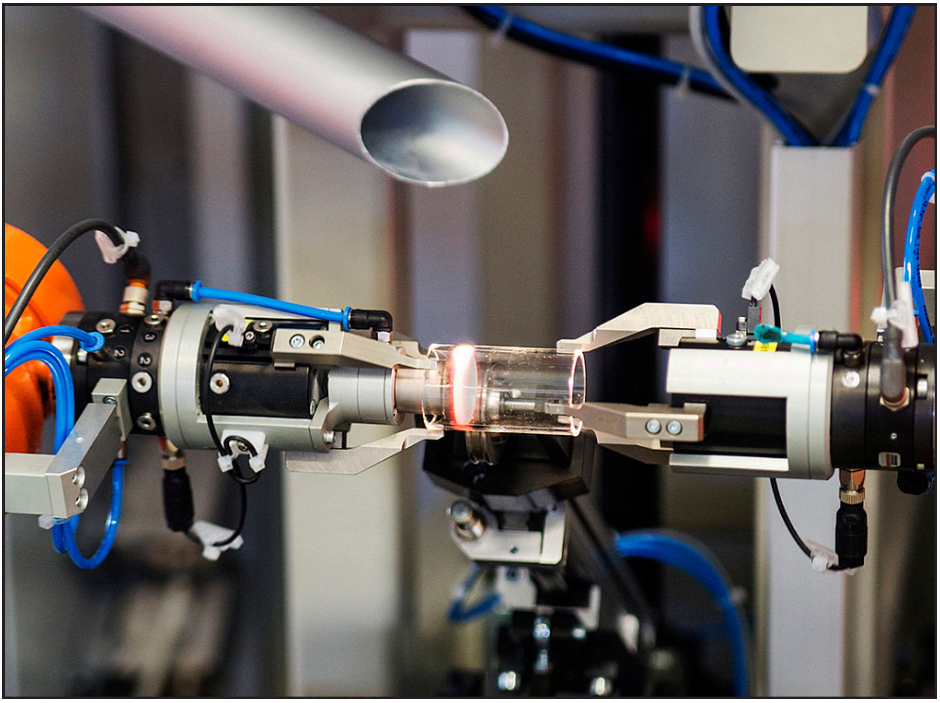
SENSITIVE AS A GLASSBLOWER

Funnels, jars and funnels - ROBU produces sintered glass filter elements.

ROBU processes, fuses and inscribes borosilicate glass automatically in one process step using a CO_2 laser and two ABB six-axis robots.

[1] <https://maschinen-insider.de/articles/4275-DE-hofmann-glastechnik-pick-place-anwendung-zur-handhabung-hochsensibler-glasroehren> Accessed June 2019.

[2] Ibid.



ROBU robots

On the fully automatic robotic and laser-based system, ROBU is working with borosilicate glass 3.3, a process that eliminates the need for conventional work with gas flames and lathes. “The trigger for the investment was a noticeable shortage of glassworkers,” explains managing director Stephan Curland. “The result was a desire to focus more on automation.”[3] The system was designed by TREBBIN together with ABB Automation and Feha Laser Tec. Feha develops and manufactures CO_2 laser radiation sources as well as optical elements for beam guidance and shaping.

The CO_2 laser radiation ensures 100% reproducibility through a targeted and controlled local heat input. In addition, the viscosity of the glass can be precisely monitored, the displayed radiation power displayed directly and the laser beam optimally controlled.

The challenge had been to develop a rotating, twelve-axis robotic assisted process. All process steps must take place on one symmetry axis, including all robot axes. This system is the first to be able to move a rotating glass tube in the hot-processed state by cooperating robots. The robots withstand the upsetting and drawing processes that you need to deform glass. At the same time they are sensitive as glassblowers. The challenge was to simultaneously move the six axes of the two ABB robots IRB 140 and the four axes of the CO_2 laser. ABB fulfills this requirement by means of absolute synchronous operation with multi-move function, infinite turning of the grippers and a high level of operating convenience. This makes it possible to address up to four robots and 36 external axes via a controller.

After concept development, ABB’s offline programming and simulation software, Robot

[3] Interview with S. Curland in May 2019.

Studio, simulated the plant's planned operations. The user is able – while production is running – to create or modify programs for new or changed parts offline, which keeps downtime to a minimum.

First, one of the robots picks up a borosilicate glass tube and the other picks up a glass filter disc, which he slides into the tube. Both robots move the work piece synchronously in rotation to a forming roller, which incorporates a constriction into the glass heated by the CO_2 laser. The robots' precision three-jaw grippers are designed for rotationally symmetrical glass components and compensate for the counter-movements of the forming roller caused by the transverse pressure to the axis as well as by upsetting and pulling movements.

In the following joining process, the CO_2 laser melts the glass filter disc which rotates with the glass tube. High-precision robots are needed for the melting process. Should the power of the laser, which is variable, be too high or too low in focus, however, the robots will change their distance to the focal point. After melting and forming, the robots move the glass tube alternately into a slightly tilted rotational position so that the laser can flatten the two sharp-edged pipe ends without any beading. The parameter ability of the laser source enables ROBU to fuse the filters into a borosilicate glass tube at high power with a single laser and label the outer walls of the glass filter crucibles with reduced power - without destroying microstructures – in a later step. ROBU uses a micro removal process developed by Feha. Then one of the robots guides the finished glass filter into the position required for laser engraving before placing it in the tray. Curland noted: "As we use several workpiece-specific grippers and forming rollers, we are able to produce glasses with different geometries, wall thicknesses, pipe outside and inside diameters, filter fineness and lengths. The control software is freely accessible to the operator. He can therefore enter different beam movements and for the ABB robot changing engraving images. The plant started operation in 2013. Amortization took place within three years."[4]

www.robuglas.com/en/about-robu/videos.html [2]

LIGHTFAB

Selective laser-induced etching (SLE) is a two-step process to produce 3D structures (also known as ISLE: In-volume selective laser induced etching — to distinguish our process from laser ablation):

In a first step, the transparent fused silica glass is modified internally by laser radiation to increase the chemical etch ability locally. To prevent the formation of cracks in the brittle material, short pulse duration (fs-ps) and a small focal volume (a few μm^3) are used. The focus is scanned inside the glass to modify a 3D connected volume with contact to the surface of the work piece. In a second step, the modified material is selectively removed by wet chemical etching resulting. Essential for the precision of the SLE technique is the selectivity. The selectivity is the ratio of the etching rate of the modified material and the etching rate of the untreated material. The selectivity in fused silica glass is larger than 500:1 resulting in long fine channels. Therefore, with the SLE-

[4] Ibid.

technique, complex 3D cavities can be produced. During the laser treatment, no material is removed but is modified in the structure. Therefore, the SLE-process is not suitable for in-line processing. The work piece must be removed from the machine to be developed in a separate bath and newly aligned for further processing. Advantages of SLE are the high precision (~ 1µm), no debris, true 3D capability and high processing speed using the micro scanners.

www.lightfab.de/files/Downloads/SLE_3D_printed_glass.pdf [2]

MICRON3DP

The 3D printing of glass combines modern and traditional techniques. One technique is the so-called FDM process, in which millimeter-thick filaments of glass are applied in layers until the object gradually emerges. The MIT as well as Micron3DP rely on the FDM technology whereby the resolution of both competitors differs. While researchers at the MIT print objects with a layer thickness of 4 mm (4000 µm), Micron3DP is able to reduce the layer thickness many times over. Their 3D printed glass objects achieve a degree of detail of 0.1 mm (100 µm) per layer. Micron3DP is currently a leader in the development of glass 3D printers. Their printers can print objects in a maximum size of 200 x 200 x 200 mm. The printers are compatible with soda-lime glass and borosilicate glass.

www.youtube.com/watch?v=ju6BtrIzz08

ARNOLD

Laser Processing

Nowadays, the reduction of production costs is one of the critical factors to survive within the global market. This also applies to the glass industry. A helpful means for achieving this target is to increase the degree of automation in the production and to reduce the labour costs respectively. At this point, the laser, successfully used in the automotive and metal industries for decades, can also be used in the glass industry.

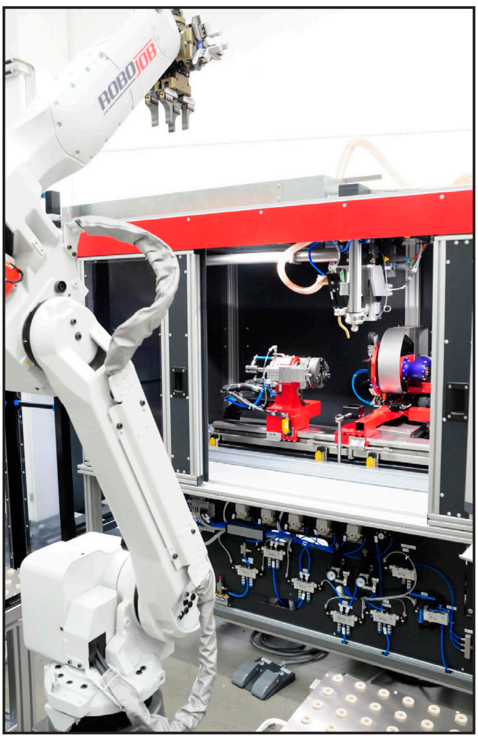
One of the important advantages is the flexibility of laser light. By focusing the laser beam, it is possible to cut quartz glass or to scratch borosilicate glass for a subsequent cracking-off process. Glass heating used for forming or joining processes can be realized by the adjustment of larger laser beam diameters on the glass. Each kind of glass, from quartz glass to AR-glass, can be processed with a laser source.

The advantages of the laser can be seen in the following summary:

- Fully automated processes
- Multiple processes with “one tool“
  – Focused cutting - defocused heating
  – Possibility of production from the tube directly, not only from tube sections
  – Low maintenance
- Temperature control during heating processes
  – Freely adjustable temperature profile, adjusted to the working process
  – High reproducibility: “constant process parameters – constant process results“

- No chemical influence on the glass during the process
  – No water condensation or soot
  – No remaining residual stresses
- No flame pressure
- No heating of the machine periphery
  – No spreading flame
  – Efficient energy introduction to the glass
  – Less temperature load of the production environment
- Adjustable intensity distribution of the laser beam
  – The heating zone at the glass can be adjusted
  – Line focus, focus point, elliptical beam geometries, etc.
- Beam guidance by mirrors
  – One laser can be used at several stations
  – During loading / unloading of one station, the laser beam can be used at another station
- Integration of the laser into existing production lines
- High cost efficiency due to flexibility, short processing times, and possible multiple use at several machines
- Low variable costs, because of low maintenance, no gas consumption, etc.

Possible glass working processes by using a laser are cutting (sublimation cutting, hot cutting, thermal shock cutting, scratching), joining of similar and dissimilar materials, forming, drilling, structuring, labelling or polishing.



ARNOLD laser robot

During a three-year research project, Arnold has provided, together with other partners, the basis for the industrial use of a laser especially for glass tube processing. Based on the following two examples, you will see the advantages of using a laser in comparison to currently available manufacturing processes.

Example for laboratory equipment manufacturing

In the field of glass tube processing, often several steps are necessary to get to the final product. In addition to forming and joining processes, cutting steps are also needed. Since these steps can currently not be integrated directly into the process chain, ready-made tubing often has to be used. However, it would be ideal to start working directly from the glass tube. Due to the traditionally manual production processes and the material properties of the glass, considerable variations of the product quality can be recognised. At this point, the advantages of a laser as an automatable tool can be used. Hence, for hot glass processes, the glass temperature can be measured by means of a pyrometer and be processed within a closed control loop. The laser receives continuously new performance data via the control so that it is possible to reach (without glass breakage) processing temperatures of glasses within seconds, which significantly reduces the overall process times.

The machine operator is able to determine the temperature profiles in testing series, which leads then to reproducible production processes. Since the glass temperature essentially follows the determined temperature due to the closed loop, the use of a temperature control is an excellent tool for stabilization of the production process, and it also establishes completely new possibilities. For example, the processing of thin-walled glass becomes possible because no flame pressure causes any deformation of the low viscosity glass.

The above mentioned intermediate steps in terms of cutting processes are possible inline between forming and joining processes due to the flexible tool laser which allows the processing of not pre-conditioned tubes.

The advantages of the laser:

- Fully automated production processes
- Short process times
- Reproducible process results

Application Example for cutting of quartz tube

Nowadays the cutting of quartz glass is typically carried out with mechanical saws. In this process, a water-cooled saw blade is operated through the quartz glass tube. Due to the tool related force application, chipping appears. Depending on further processing, the cutting edge has to be reworked, which includes grinding, fire polishing, etc.

In any case, washing and drying of the tubes is required to remove the chips. In contrast, no additional process steps are required for laser cutting of quartz tubes.

Depending on the wall thickness, the laser beam is focused and is used to evaporate the irradiated material. The cutting width is about 100 microns. The contamination of the glass tube due to occurring quartz smoke can be avoided by an efficient suction. The cutting time for tubes with outer diameters up to 50 mm and wall thickness up to 3 mm takes only a few seconds. The separated quartz tubes can be directly taken out after the laser cut. Post-processing steps, such as in current production, are not necessary.

The advantages are as follows:

- One-step process
- Fully automatable
- Short process times
- No need of machines for cleaning and drying
- No need of water treatment
- No tool wear, compared to cutting blades
- Constant cutting results
- Automatic process adjustment to different tube geometries

NOTES

In order to perform customer inquiries and application tests and for carrying out complete test series, two modifiable glass working lathes for tube diameters from 3 mm up to 160 mm are available at ARNOLD. These systems are coupled with lasers in the power range from 60W to 2500W. The installation of new test equipment (also for flat glass, plastics, etc.) is possible. In addition, ARNOLD works closely together with well-known research institutes in order to develop optimal solutions for their customers.

If you are interested in a demonstration of glass processing with laser radiation or you are already thinking about concrete applications within your product portfolio, please feel welcome to visit ARNOLD in Weilburg. They are looking forward to performing some tests with your components.

www.arnold-gruppe.de [2]

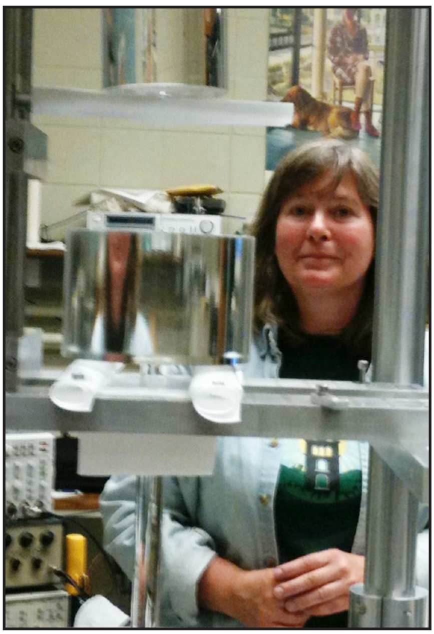
Silica and the Gravitational Wave

by
Sally Prasch*

ABSTRACT

A century after Albert Einstein predicted his general theory of relativity, we have detected gravitational waves. In this presentation, I will be talking about how silica played an important part in the techniques that allowed the Laser Interferometer Gravitational-Wave Observatory (LIGO) to achieve a length precision that is 10,000 times smaller than a proton. I will also be talking about the work that I did for Dr. Steve Penn who significantly reduced the thermal noise in fused silica. Dr. Penn was among those involved with the LIGO research who were awarded a "Breakthrough Prize: Scientists Changing the World" medal lauding the landmark research.

I have worked as a scientific glassblower since 1970. I apprenticed in junior high and high school with Lloyd Moore, earned a BFA in Ceramics and Glass from the University of Kansas, an Associate degree in Applied Science and a Certificate in Scientific Glassblowing from Salem Community College. I have worked in industries, universities and have run my own business. I have a long history with glass, having seen many experiments and wonderful discoveries. I have seen the changes in what is being made by scientific glassblowers, and I continue to flow with the ever-changing science and the ever-changing fabrication of glass.



Me praying that the silica would not fall. It is hard to see but everything is suspended by silica fiber.

For some of you who have not worked in the university setting, I would like to describe what I find it to be like. It is different from industry where things are sometimes bigger than you think they should be and sometimes repetitive. In industry, most of the time I was working from a blue print. Things were well thought out and signed off by engineers. At universities, people come into the glass shop with ideas floating through the air that challenge me daily. They may not have a drawing or maybe they have something on a napkin with things like "put a large tube here." They of course also come in with mundane repairs but the majority of my work, which is not just chemistry apparatus, can be a little weird. I am working for geology, biology, earth science, polymer science, engineering, physics and many other departments. You would think after 49 years that I would have seen it all, but no, not at all.

People from all over the world are attending universities, making a rich diverse culture that I love. All these people bring with them differ-

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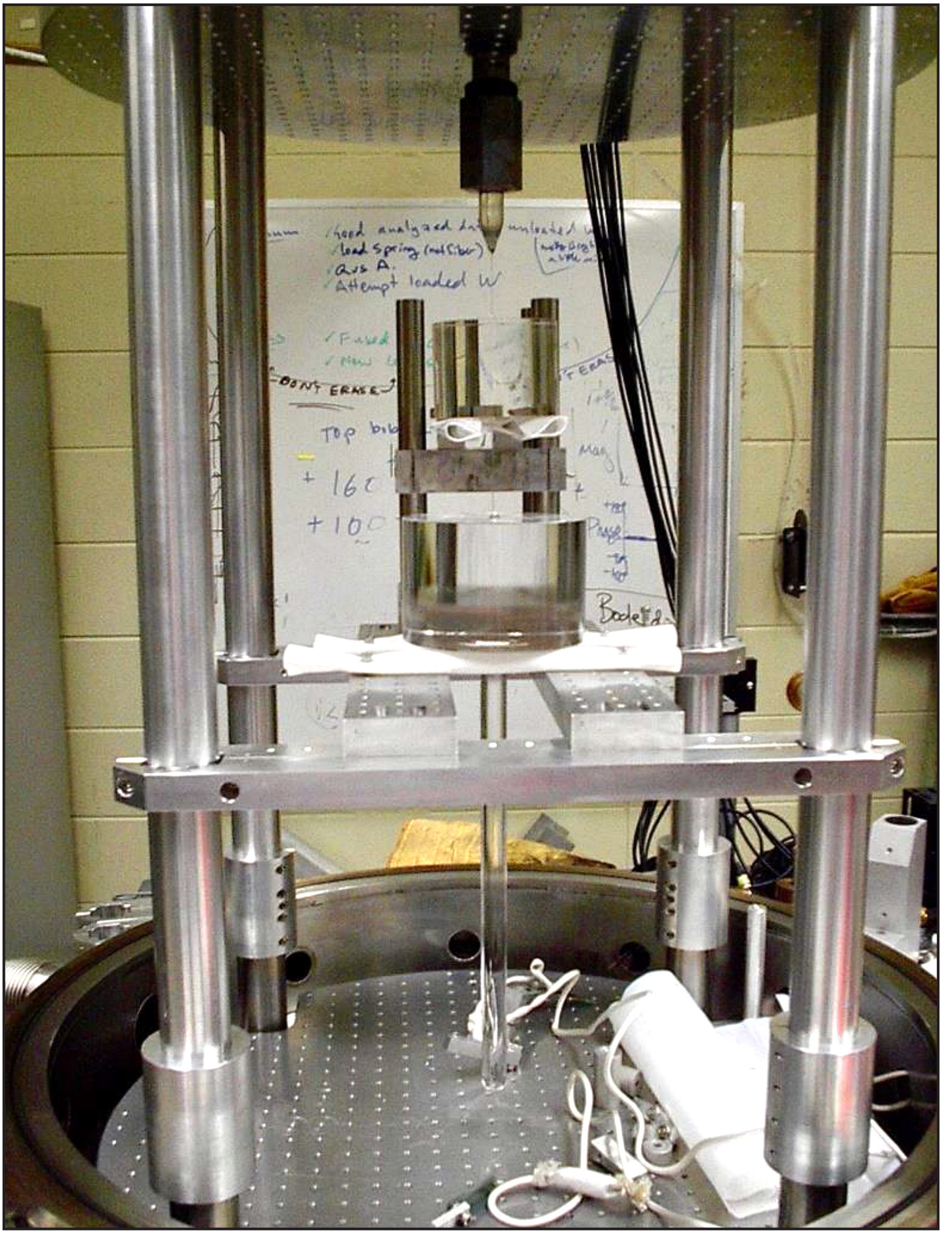


Quartz suspended in open vacuum chamber.
In this photo there is Teflon™ under the quartz in case it falls during the set-up procedure.
The Teflon™ is removed before the chamber is evacuated.

ent ideas and different ways of working. As a university scientific glassblower, I need to understand my clients' culture, how they work and when they work their best. Language can sometimes be a problem, but the love of science brings us together and I continue to make wild things for some of the most intelligent people on earth. So, what I am saying is that people from all over the world come in with ideas that I have never seen before and say "Can you make this for me?" or "I need this to do my science and I need you to make this."

This is usually followed by "I need this right away." I find myself reaching way down, reaching way down into my soul for answers to quickly make what they need to help them.

Steve Penn works at Syracuse University in the Physics Department. To look at him, you would never think that he is a world known scientist working on what will be known as one of the biggest discoveries in my lifetime. Before the discovery of gravitational waves, not everyone believed that they would ever be found, and funding at that time was hard. Often a scientist shares their mind-blowing research with you, but not all of it works out or it may be something big like this.

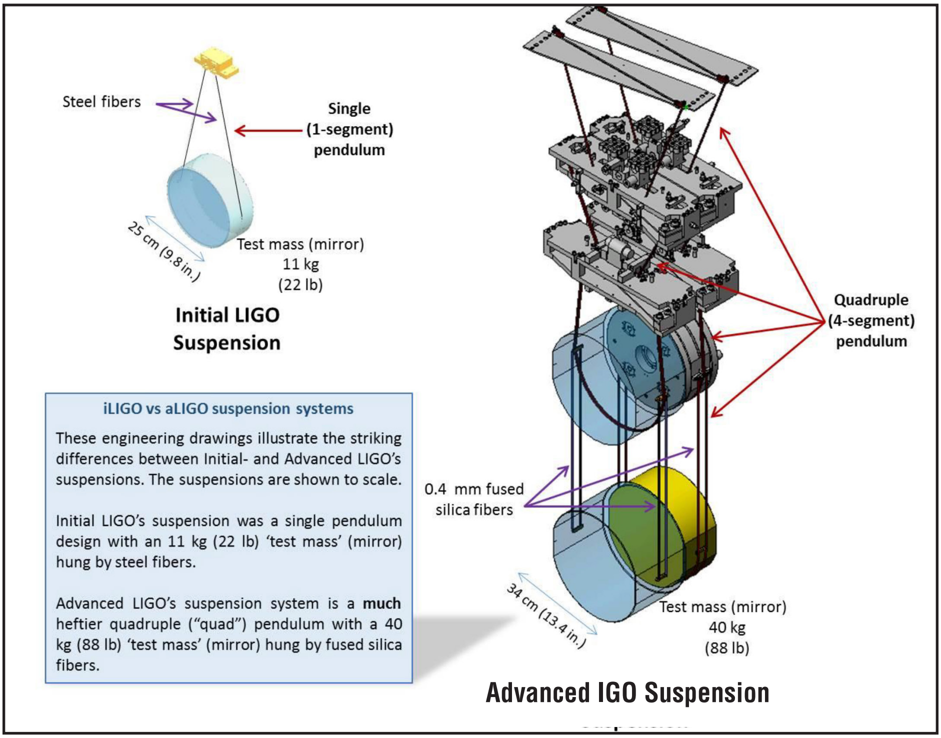
Steve Penn, Syracuse University, Hobart and William Smith Colleges, currently chairs the LIGO Scientific Collaboration Coating Working Group

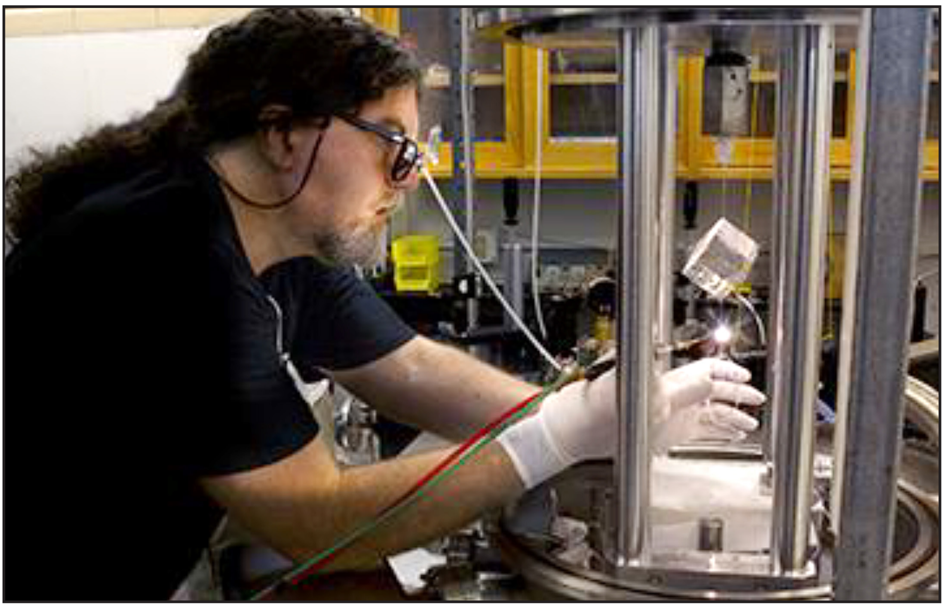
One day, Steve walked in and informed me that he had a very pure 20 lb sample of fused silica that cost a lot of money and that he wanted me to hang from a fiber optic inside a vacuum chamber. I was like, "I don't think so;" to me it just seemed to be too heavy. He interrupted my disbelief and said "Sally, you can do this, I calculated it all out." I work with really smart people and they are usually correct. So I started working on something that turned out to be one of the biggest highlights of my life.

I first had to put a nub of the same make of fused silica onto the large solid pieces. The nubs, about 8 mm x 8 mm, would be the material used to pull the fiber. The pieces were then annealed before suspending them with Teflon™ in a large stainless steel vacuum chamber. Movement or vibration would interfere with this experiment, so the vacuum chamber is located in the sub-basement of the Physics Building on a table designed to isolate it from ambient vibrations. With a small torch, I heated the nub and then pulled quickly up and attached it to another piece of silica. I repeated this, eventually clamping it in a chuck at the top of the vacuum chamber. We then removed the Teflon™ supporting the large pieces of silica, leaving them suspended. With the vacuum chamber evacuated, Steve pinged the silica and it resonated for three months. When he told me this I was amazed, but what he said amazed me more. He looked at me and said "We need to find a better material, but this is the best we have for now." I think about how much money and time goes into many parts of finding gravitational waves, it is so incredible. Research continues and we will find new ways to see more.

Gravitational waves are ripples in the curvature of space-time caused by massive objects moving with extreme speed. This allows us to observe black holes and other massive objects in the distant Universe.

To detect these waves, laser light beams travel back and forth down 4 foot tubes that are kept under a near perfect vacuum. The beams are used to monitor the distance between





Steve Penn hanging fused silica from fiber

mirrors that are precisely positioned at the ends of the tubes. Guess what they are made of? You got it, fused silica with a TiO_2 +Ta_2O_5 coating. The distance between the mirrors will change when a gravitational wave passes by the detector. A change in the length of the arms smaller than one-ten-thousandth the diameter of a proton (10^{-19} meter) can

Teaching the happy gravitational wave crew how to hang silica discs

be detected. The reflection off mirrors and the dynamics of that reflection are incredibly important if you want to precisely know the mirror's position. Steve Penn's research significantly reduced the thermal noise in the fused silica and the coating.

The Laser Interferometer Gravitational-Wave Observatory (LIGO) would never have seen a gravitational wave without this research. Thousands of people all over the world have worked hard on this project and I am very happy to be one of them.

(www.youtube.com/watch?v=B4XzLDM3Py8)

Use of Synthetic Fused Silica in the Growth of Ultra-high Purity NaI(Tl) Single Crystals

by

Burkhant Suerfu, Ph.D.*

ABSTRACT

Dark matter is an unknown form of matter that comprises about three quarters of all matter in our Universe, and its existence and nature is one of the most crucial questions of this century. It has been hypothesized that dark matter can occasionally collide with an atomic nucleus, and the recoiling nucleus will further ionize nearby atoms and molecules. While most direct detection experiments have reported null results, for over a decade the DAMA/LIBRA experiment has been seeing an annually modulating event signal consistent with dark matter in high-purity thallium-doped sodium iodide (NaI(Tl)) crystal scintillators. To make a model-independent test of the DAMA/LIBRA annual modulation, NaI(Tl) single crystals with even higher purity are required. Recently, we have successfully grown ultra-high purity NaI(Tl) single crystals. In this process, the use of special quartz glass has been essential. In this paper, we detail the processes used to grow the crystal and examine the close connection between synthetic fused silica glass material and the growth of ultra-high purity NaI(Tl) crystals.

INTRODUCTION

Modern astronomical and cosmological observations indicate that about three quarters of all matter in our Universe is comprised of dark matter, a form of matter that is only known to interact via gravity. [1, 2, 3] However, the existence of dark matter has never been directly confirmed in a laboratory. In addition to gravity, certain dark matter candidates—weakly-interacting massive particles (WIMPs)—are also hypothesized to interact weakly with nucleons in atomic nuclei. [4] For WIMP particles with masses of a few GeV, the interaction with atomic nuclei is manifested as nuclear recoils, and the recoiling nuclei can further ionize nearby atoms and molecules. As a result, most ground-based dark matter direct detection experiments are searching for the ionization effects of dark matter-induced nuclear recoils.

Since dark matter detectors are often also sensitive to ordinary ionizing radiation, dark matter direct detection experiments are usually challenged by the radiation backgrounds from cosmic rays, radioactivity in the ambient environment and in the detector medium itself. Therefore, these detectors are often deployed underground to block cosmic rays and shielded to reduce gamma-rays and neutrons from the ambient environment. Furthermore, to lower background from radioactive isotopes and impurities, the detector is often constructed from materials of very high radio-purity.

Additionally, annual modulation in the event rate can also point to the existence of dark matter. [5] Such annual modulation is expected from the yearly change in the relative velocity between the Earth and the dark matter particles, as the Earth revolves around the Sun which is moving with constant velocity in the bath of dark matter particles in the Milky Way. This scheme is illustrated in Figure 1.

Over the past decades, dark matter detectors have advanced significantly in both size and purity, yet no experiment has observed dark matter interaction with the exception of the DAMA/LIBRA experiment. [6] Using a matrix of high-purity NaI(Tl) crystal

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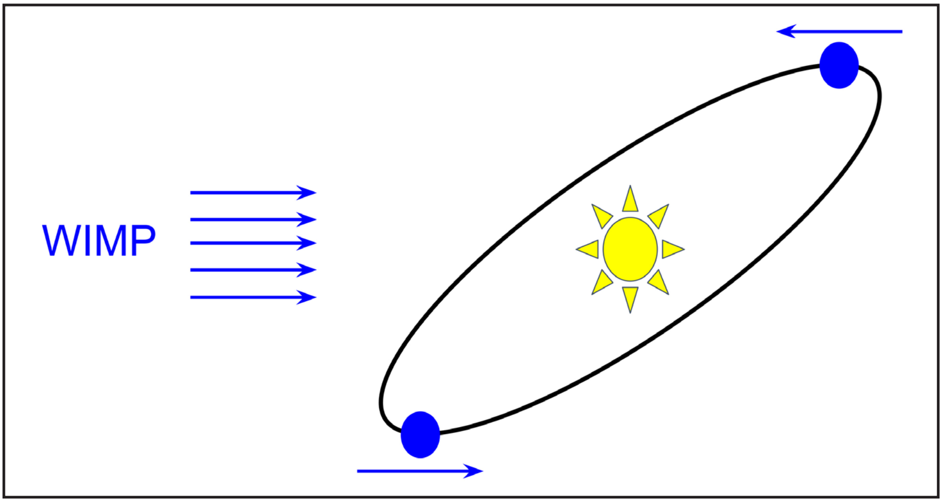


Figure 1: *Illustration of dark matter annual modulation. When the Sun moves in the Milky Way, it is seeing a constant wind of dark matter particles. As the Earth revolves around the Sun, the relative velocity between the Earth and the bath of dark matter changes on a yearly basis. This is manifested in dark matter detectors as an annual modulation in the event rate.*

scintillators, the DAMA/LIBRA experiment observed in 2-6 keV energy window an annually modulating event rate of approximately 0.01 cpd/kg/keV on top of a constant background of about 1 cpd/kg/keV. [7][1]

To make a model-independent and definitive test of the DAMA/LIBRA annual modulation efficiently, NaI(Tl) single crystal detectors of even higher purity are desired. Currently, several NaI(Tl)-based dark matter experiments are running, but none of these experiments have crystals with purity comparable to those used in the DAMA/LIBRA experiment. [8, 9] In dark matter searches, a major impurity of concern in NaI(Tl) crystals is ^{40}K, which can decay by the capture of a K-shell electron about 11% of the time with an emission of a 1.46-MeV gamma-ray. Should the gamma-ray manage to escape the crystal, the subsequent 3-keV atomic de-excitation will appear in the middle of the region of interest (4-6 keV). The K level in the DAMA/LIBRA crystals is about or below 20 ppb whereas in other experiments the average K level in the final crystal is approximately 32-42 ppb. [6, 8, 9] [2]

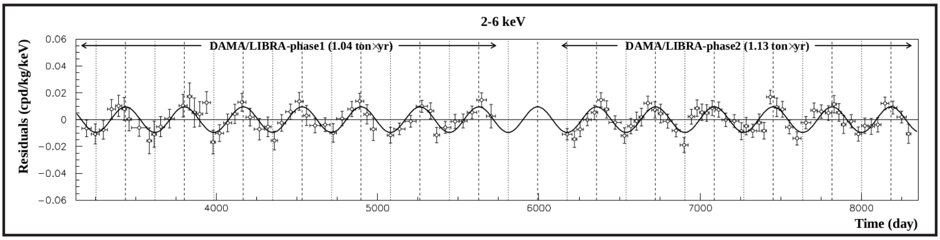


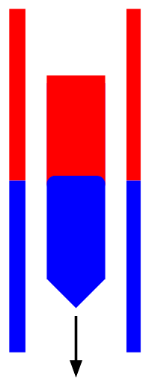
Figure 2: *DAMA/LIBRA annual modulation in the 2-6 keV window. [7] The modulation has an amplitude of about 0.01 cpd/kg/keV on top of a constant background of approximately 1 cpd/kg/keV.*

[1] cpd is short for count per day.

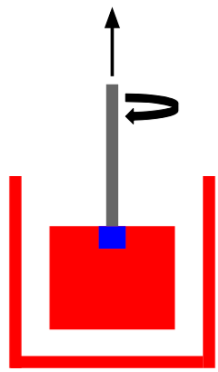
[2] The natural abundance of 40K is about 0.01%, and in many cases results are reported in terms of 39K.

CRYSTAL GROWTH BY THE VERTICAL BRIDGMAN METHOD

Industrially, large alkali halide single crystals are often grown from melt using either the Bridgman or the Kyropoulos method. [10, 11] In the Bridgman method (Figure 3a), the raw material is placed inside a crucible with a tapered end, melted and slowly cooled from the tapered end. During the cooling, spontaneous crystallization at the tapered end provides a nucleation site for the subsequent part of the crystal. In the Kyropoulos method (Figure 3b), a seed crystal is brought into the molten raw material. As the seed crystal is slowly rotated and pulled up, the melt crystallizes around the seed crystal into a single crystal. Although the Kyropoulos method has a higher yield, the process is complicated, and the rotation and translation of the seed crystal make it hard to completely seal and protect the melt from the ambient environment. In this regard, the Bridgman method has an advantage as it is possible to completely seal the raw material inside the crucible.



(a) Crystal growth by the vertical Bridgman method. A crucible containing molten raw material is slowly brought from a hot zone (red) into a cold zone (blue).

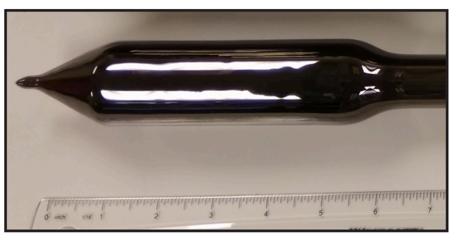


(b) Crystal growth by the Kyropoulos method. A seed crystal (blue) is slowly rotated and pulled out from the melt.

Figure 3: *Illustration of the vertical Bridgman method and the Kyropoulos*

Fused quartz glass is often used as a crucible material for crystal growth as it offers good high-temperature performance, chemical resistance, and is readily available in very high chemical purity. However, in the growth of NaI single crystals, quartz is not suitable as molten NaI, or more specifically the trace amount of NaOH found in NaI, can attack the

(a) Photo showing NaI crystal sticking to uncoated quartz crucible.



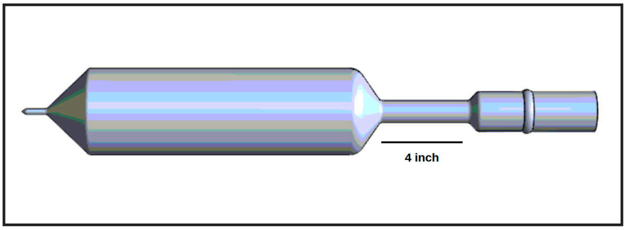
(b) Photo of a 1-inch-diameter quartz crucible coated with a layer of pyrolytic carbon.

Figure 4: *Photos of NaI crystal sticking to uncoated quartz glass and a quartz crucible coated with pyrolytic carbon*

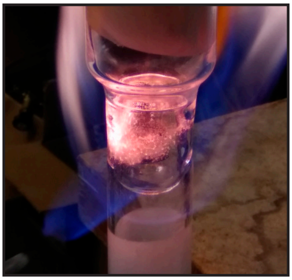
quartz glass, causing the crystal to stick to and fuse with the quartz crucible (see Figure 4a). [12, 13, 14] Subsequently upon cooling, a mismatch between the coefficient of thermal expansion causes the crystal or the crucible to crack. Fortunately, it has been shown that the sticking can be alleviated or prevented by applying a layer of pyrolitic carbon coating (Figure 4b) as a physical barrier and a getter. [15]

CRYSTAL GROWTH PROCEDURE AND PURITY MEASUREMENT

To grow a NaI(Tl) single crystal by the vertical Bridgman method, dry Astro-grade NaI powder from Sigma-Aldrich is mixed with TlI powder and loaded and sealed inside a 4-in.-diameter, 2-ft-long carbon-coated quartz crucible. The Astro-grade NaI powder is chosen because it already has purity comparable to or higher than the NaI crystals used in the DAMA/LIBRA experiment. A CAD rendering of the crucible is shown in Figure 5a. Prior to coating,[3] the quartz crucible is cleaned at Seastar Chemicals with a mixture of Detergent 8, hydrofluoric acid (HF) and deionized water at room temperature, and then soaked with a mixture of ultra-high purity HF acid and nitric acid (HNO_3) for 1 hour. After the carbon coating, the crucible is cleaned with ultra-high purity hydrochloric acid (HCl) at Seastar Chemicals. The NaI powder is thoroughly vacuum-baked and dried at increasing stages of temperatures over the course of two weeks. [14] After the NaI and TlI powder have been loaded into the crucible, the crucible is sealed by fusing the quartz wall with a quartz plug under vacuum (Figure 5b).



(a) CAD rendering of the quartz crucible used to grow NaI(Tl) crystals. The right end is 45×48 quartz tubing for connection to vacuum adapters.



(b) Sealing of the crucible.

Figure 5: *CAD rendering of the quartz crucible used to grow NaI(Tl) crystals and photo showing the process used to seal the crucible. The seal of the crucible is achieved by heating the 1 inch.-diameter neck of the quartz crucible and fusing it against a quartz plug, which is held vertically in place by a bump on the neck. During the process, the inside of the crucible is kept under vacuum.*

After the seal, the crucible is taken to Radiation Monitoring Devices (RMD) at Watertown, Massachusetts, where it is heated to above the melting point of NaI (661°C) for three days in a vertical Bridgman furnace to thoroughly mix the NaI and the TlI. Subsequently, the crucible is slowly brought down through a thermal gradient. After the entire crucible has entered the cold zone of the furnace, the furnace temperature is slowly lowered to room temperature over a week. Subsequently, the quartz crucible is cut open with a diamond wire saw, and the ingot is cut to the final detector geometry and polished. A photo of a typical crystal ingot is shown in Figure 6.

During crystallization, certain types of impurities are repelled from the crystal matrix whereas other types are attracted into the matrix. The degree of separation is character-

[3] For the crystals in this study, the carbon coating was performed by Sandfire Scientific.

ized by the equilibrium distribution coefficient K, defined as the ratio of impurity concentration in the liquid phase to that in the solid phase in equilibrium. [12] To estimate the overall K concentration in the entire crystal, K concentrations at three different locations are measured and the distribution is fitted against the model. [14] The K measurement is carried out with inductively-coupled plasma mass spectroscopy (ICP-MS) at Seastar Chemicals. To perform reliable and accurate measurement, the spectrometer is calibrated with a NaI powder sample that has been pre-assayed with high-purity germanium (HPGe) detectors underground at Laboratori Nazionali del Gran Sasso (LNGS) for three months.

Figure 6: *Photo of a typical NaI(Tl) ingot obtained with vertical Bridgman method and a carbon-coated quartz crucible*

PURITY OF THE QUARTZ CRUCIBLE

Of different grades of fused quartz glass, synthetic fused silica offers the highest purity since it is made from purified silicon-containing precursor chemicals while ordinary quartz is usually made from naturally occurring sand or quartz mineral. Table 1 shows the difference in chemical purity between regular quartz and synthetic fused silica. However, most physical and chemical properties of synthetic fused silica is identical to regular fused quartz, and their difference is emphasized mostly in the context of optics where less impurities in synthetic fused silica offers improved transmittance for UV light.

Element	Concentration (ppm)	
	GE 214	Suprasil 310
Li	0.6	<0.01
Na	0.7	<0.05
K	0.6	<0.01

Table 1: *Typical impurity concentrations of GE 214 fused quartz [16] and Heraeus Suprasil 310 synthetic fused silica [17]*

To study the influence of the purity of the quartz crucible, two crystals were grown, one in a crucible made of regular quartz glass while the other in a synthetic fused silica crucible. All other handling procedures were kept identical. The impurity measurement on the resultant crystal is carried out as described in Section 3, and the result is shown in Figure 7.

Compared to the raw powder which has a K concentration of 8±2 ppb and a Li concentration at an upper limit of 10 ppb, the results indicated that the crystal grown in the ordinary quartz crucible showed an increase in K and a significant increase in Li. However, the crystal grown in the synthetic fused silica crucible showed no change in

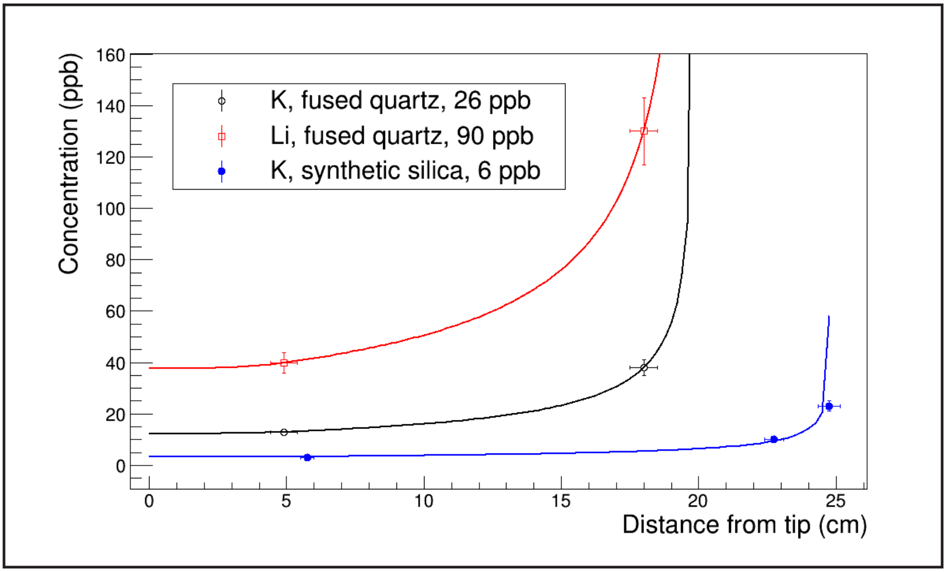


Figure 7: *Impurity distributions in crystals grown in a regular fused quartz crucible and a synthetic fused silica crucible. The crystal grown in a regular quartz crucible experiences a moderate increase in K and a significant increase in Li.*

K, and Li concentrations were all below the detection limit of 10 ppb, indicating that the impurities are introduced into the crystal during growth through diffusion from the crucible material. As the rate of diffusion increases exponentially with temperature, the elevated temperature at which the raw material melts (above 661°C) and the prolonged contact time between the melt and the crucible required for single crystal growth (on the order of months) have strengthened the impact of impurity diffusion. The change in Li, in particular, supports this point since Li is present only in regular fused quartz (Table 1), and it has a higher mobility therefore a higher rate of diffusion than K is expected. [18]

CONCLUSIONS

Single crystals of ultra-high purity play an important role in the direct search of dark matter and other low-background experiments. Yet the growth of ultra-high purity crystals are challenging due to various ways contamination can be introduced in the process. In this article, we have outlined a method to grow NaI(Tl) single crystals using quartz crucibles and the vertical Bridgman method. Furthermore, purity measurements on crystals grown in a standard quartz crucible and a synthetic fused silica crucible indicate that impurities found in the quartz crucible are capable of diffusing out from the crucible and contaminating the final crystal. This suggests that the use of fused silica of synthetic origin is critical in the growth of such ultra-high purity crystals.

ACKNOWLEDGEMENT

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The Importance of Exploratory Research and Serendipitous Discovery

by

Jesse Kohl, Ph.D*

ABSTRACT

While computer modelling and simulation capabilities have greatly accelerated scientific research, many of the truly disruptive innovations have been made through serendipitous discovery and hands-on exploratory research in the lab. Value and use of this hands-on approach must continue into the future for significant new discoveries to be made. This article presents examples of the serendipitous nature of research and lessons in innovation from the career of one of Corning's great pioneering researchers, Dr. James Franklin Hyde, "The Father of Silicones."

We are now living in an exciting age where there is ever increasing ability to simulate and predict the outcome of scientific experiments. It is for this reason that I was compelled to speak at the 2019 ASGS Symposium about the importance and continued need for hands-on exploratory research in the lab and serendipitous discovery, which perhaps not surprisingly is still the mother of many inventions. During my lecture, I shared some lessons of discovery and innovation from Corning's past as well as some that I have learned during my four years at the company. My hope was that doing so will enhance your own paths to new discoveries, and encourage you to take the necessary risks sometimes required to make pioneering breakthroughs in your own respective fields of work. For this paper, I have chosen to focus on one Corning's great inventors, Dr. James Franklin Hyde, "The Father of Silicones." [1-4] Dr. Hyde's work teaches us that sometimes great inventions sit idle on the shelf for many decades for a problem to come along for them to solve. Further, his achievements demonstrate that through vision, tenacity, and serendipity, new and unknown possibilities can be realized.

The year was 1930 and Dr. Eugene Sullivan, eminent glass chemist, Vice President and Director of Research at Corning Glass Works, was thinking about a threat to the corporation, plastics. The plastic industry was still in its infancy, but already it was clear to Dr. Sullivan that one day, perhaps in the not-so-distant future, plastic products might replace their glass counterparts, with grave consequences for glass makers such as Corning. [1-3] As he pondered this sobering prospect, Dr. Sullivan began to formulate an idea: what if it were possible to combine the advantages of glass with those of plastics to produce materials that were stronger and more heat-resistant than plastic, but more flexible than glass? [1, 2]

No one had attempted to make such hybrid materials that Sullivan envisioned, and therefore, to enter this area was a leap into an uncharted realm of materials science. Because of the great depression, funding was scarce, and resources were unavailable. The general consensus at the time was to maintain the status quo and weather the storm. However, to Dr. Sullivan the question persisted, "what if?" Guided only by intuition, he and Corning forged ahead with the vision of glass-plastic hybrids and made a most unorthodox decision for a glass company, the hiring of organic chemist Dr. Frank Hyde. [1]

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Frank, who was born in 1903, always had a curiosity about what things were made of and what made them tick. [2] Growing up in the small village of Solvay, New York in the vicinity of a huge industry, The Solvay Process Company (a source of baking and washing soda) greatly increased his interest in chemistry. [1]

In the fall of 1919 as a freshman at Syracuse University, Hyde had his first view of a real lecture hall and laboratory, both of which he thought were wonderful. During the twenties, Frank had the opportunity to learn from some of the leading teachers and scientists of the time: Professors Ross Baker and R. S. Boehner of Syracuse U., and Professor Roger Adams of the University of Illinois where Frank received his Doctorate of Philosophy in 1928. [1]

His good fortune continued when he was awarded a two-year post-doctoral fellowship to work with Professor James B. Conant of Harvard University. Dr. Conant had expertise in both organic and in physical chemistry and was the President of Harvard from 1933 to 1954. In addition, between 1941 and 1946, he was the chairman of the National Defense Committee, and with his friend Vannevar Bush, he helped ramp up the Manhattan Project that led to the creation of the atomic bomb. In short, Frank had superb mentors. [1]

Early in 1930, Frank returned to Cambridge after an interview with the Dupont Chemical Company and met Professor Conant in the hallway of the Converse Laboratory. The Professor inquired "How did you make out?" Frank replied that he was offered a job as a research polymer chemist at a salary of $3,200 a year. Conant shot back, "They should do better than that! While you were gone Bill Taylor of Corning Glass Works was here looking to hire an organic chemist for their research laboratory." Frank exclaimed, "What do they want with an organic chemist in a glass factory?" Conant replied, "I don't know," and in his usual curt manner quipped "Why don't you go down to Corning and find out?" [1]

Here again, Frank was in the right place at the right time. It had occurred to him that Corning could possibly be interested in organo-silicon chemistry.

Frederic Stanley Kippling's work in the early part of the 20th century had indicated that silicon atoms asymmetrically substituted with organic groups would show optical activity as in carbon chemistry. Frank journeyed to Corning and interviewed with Dr. Sullivan and Mr. Taylor. They discussed the field of organo-silicon chemistry, and Frank accepted the offer to begin work in Corning's research laboratories. In September 1930, Frank began making organo-silicon compounds and comparing them with glass and plastic products.

At that time, Corning was working to develop glass fibers for use in electrical insulation. To provide effective insulation, tapes required a resin to fill up the air-space between fibers, since air pockets caused breakdowns and short circuits. Glass fiber tapes were more heat resistant than cotton and would allow the operation of electrical equipment at higher temperatures, and at higher speeds than ever before.

Hyde's first experiments with organo-silicon compounds at Corning thus involved the pursuit of a superior resin filler for use in glass-fiber electrical tape. He began by preparing two compounds with silicon-carbon bonds, combining the two basic elements of glasses and plastics. When he hydrolyzed the compounds, glass-like brittle solids were created which liquefied when heated in a Pyrex® test tube. Frank continued to heat the glass tube beyond its melting point until the tube became so hot, it sagged and elongated, but the products in the tube remained unchanged. What resulted was a clear liquid with remarkable thermal stability. This liquid product with a silicon-carbon bond was the first example of the hybrid materials that Dr. Sullivan had sought. It was the first compound that combined the basic components of glass and plastic, the first crude laboratory sample of organo-silicon polymers now known as silicones. [1-3] (Photo 1)

Photo 1. *Dr. Frank Hyde with assistant pictured in 1945 at Corning Glass Works. [4]*

As Frank continued his research, he was frequently called upon to solve industrial problems. In 1932, Corning was working to solve a structural problem involving a fifteen by forty foot window being installed over the entrance of the RCA building in New York City's Rockefeller Center. Corning had prepared twenty by thirty inch blocks of Pyrex® glass which would form the larger window. A mortar was needed to help cement the glass blocks in place and prevent surface stress. Hyde developed a mortar by dissolving the molecules of a monomeric compound in solution, reasoning that the monomer would polymerize over time. The mortar would then adhere to sheets of resin placed between the glass blocks and form one piece of vinyl acetate that would hold the blocks in place. [1]

With special dispensation from the Glazers Union, Hyde spent a month putting the glass blocks in place, wedging the vinyl acetate sheets between the blocks and using the mortar that he had developed to cement the blocks in place. His reasoning was sound, the monomer did polymerize over time, and the window project was a success!

In 1934, Corning's Fiber Products Division focused on the development of woven insulating tapes and fire-proof draperies. Hyde directed some of his work to these products. His research resulted in the development of a resin for glass-fiber tapes, designated as 990A resin.

By this time, Owens-Illinois had invented a way of drawing fibers from an electrically heated boat with many small orifices. Since Corning held patents in the fiber area, a cross-licensing agreement was arranged between the two companies that eventually led to the joint venture, Owens-Corning Fiberglass Corporation. Now, the binders first used to create household fiberglass insulation were prone to attack by bacteria, fungus, and other biologicals. This caused deterioration of the product. Working with the Bakelite

Corporation, Hyde developed a water-soluble, partially-polymerized, phenol-formaldehyde resin with built-in germicidal, fungicidal, and insecticidal properties to combat this problem. However, when Frank applied this binder to the fiberglass, he noticed that the product turned pink. Today, Owens Corning Fiberglass continues to use The Pink Panther in its advertising.

With the onset of World War II, Hyde's work on glass fibers for electrical insulation soon gained a powerful ally, Rear Admiral (then Commander) Hyman Rickover, who was interested in thermally stable insulation for submarine motors that could operate at a temperature of 250°C or higher. If able to run at higher temperatures, motors could be smaller and still deliver the required horsepower. Rickover could see the potential use for fiberglass insulation in submarines, but without varnish or resin to protect the wires, they were vulnerable to corrosion and abrasion due to vibration. When Rickover was shown a piece of glass tape coated with varnish made from Hyde's 990A resin, he examined the tape and said "Now you've got something, I want it tomorrow!" (Photo 2)

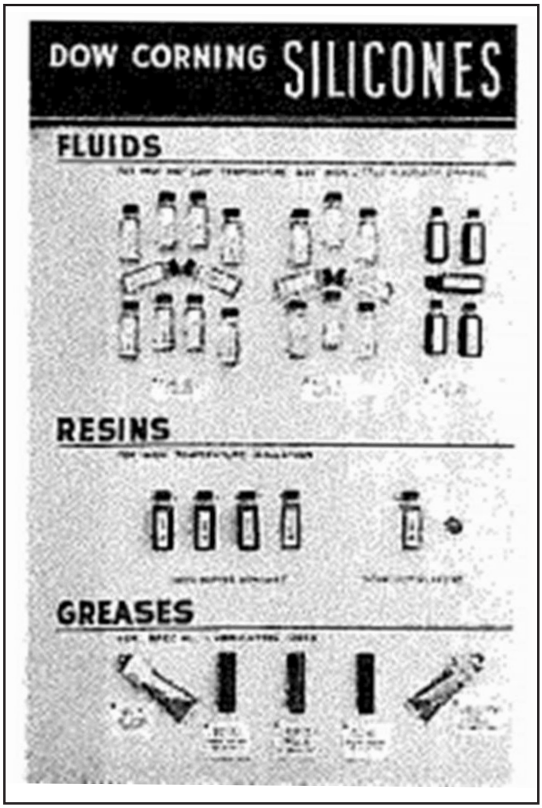

Photo 2. *Early Dow Corning Advertisement*

Out of Hyde's research and the demand from the U.S. Navy, the Dow Corning Corporation was formed with a handshake in 1942. [4] Dow Chemical had the magnesium and the halide intermediate needed for Hyde's Grignard synthesis of silicones that Rickover wanted, and Rickover could secure the steel and other materials that Dow Corning required to build their first silicone manufacturing plant in Midland, Michigan. Ironically, the Navy never used a motor that incorporated Dow Corning silicones on submarines during World War II. The first product to emerge from Dow Corning for use in the war was a sealing compound for spark plug wells on U.S. Army airplanes to prevent arcing under high altitude, humid conditions over the North Atlantic and North Sea. [1]

By using new methods of synthesis, polymerization, and co-polymerization developed by Hyde, polymers of very different characteristics were produced. In addition to his scientific successes, Hyde was also responsible for moving silicones from the laboratory, where they were of academic interest, to commercial fields where they were of interest to many diverse enterprises. [1-5]

As if Dr. Hyde's career and accomplishments were not impressive enough, it turns out that it was one of his discoveries that laid dormant for over thirty years that enabled a significant breakthrough in optics, the development of low loss optical fiber for communications. And Hyde's discovery, made in 1934 was a fundamentally new way to make glass. [1-3]

At the time when Dr. Hyde began working at Corning, glass was traditionally made by melting dry mineral ingredients. Making a pure silica glass required melting the purest quartz sand at temperatures exceeding 2000°C with oxygen-hydrogen burners or by electric arc furnaces.

Hyde's unprecedented idea was to synthesize the silica glass from liquid chemicals instead of the conventional powders. In a crude experiment, he sprayed silicon tetrachloride liquid into the flame of a welder's torch. It reacted with the water vapor that was produced by the burning of the fuel to form an extremely pure glass, now called fused silica. [1-3] (Photo 3)

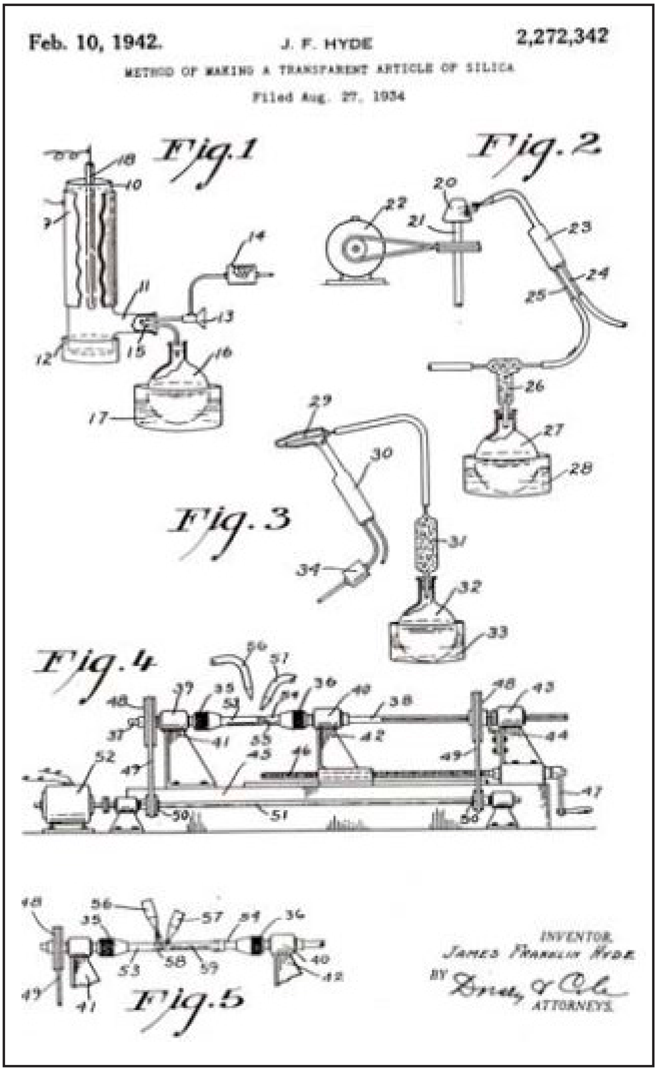

Photo 3. *Hyde's flame hydrolysis process to make fused silica from silicon tetrachloride vapor [6]*

This process, which Frank patented in 1934 [6] called flame hydrolysis, created impurity-free silica in the form of fine powder that he referred to as 'soot.' This soot could be pressed into various shapes and sintered, enabling the production of high purity fused silica telescope mirrors and spacecraft windows. Later, fused silica also made the miniaturization of computer chips possible, as it is used in high transmission photolithography-masks and lenses. [1-3] (Photo 4)

It was not until July 1967 when Rutgers graduate Dr. Peter Schultz joined Corning, and under the direction of research director Bill Armistead, was charged with taking a 'fresh look' at Hyde's flame hydrolysis process to see what else could be done with this tech-

Photo 4. *Fused Silica Boule Made by Hyde's Flame Hydrolysis Furnace Process [7]*

nology. And in collaboration with Dr. Don Keck and Dr. Bob Mauerer, they eventually achieved an optical fiber with a loss of only 16 dB/km using Frank Hyde's flame hydrolysis method. [7] They filed their discoveries in 1970, disclosing the use of doped fused silicas and the method of making these fibers by depositing the core glass on the inside of a cladding tube. [7] (Photo 5).

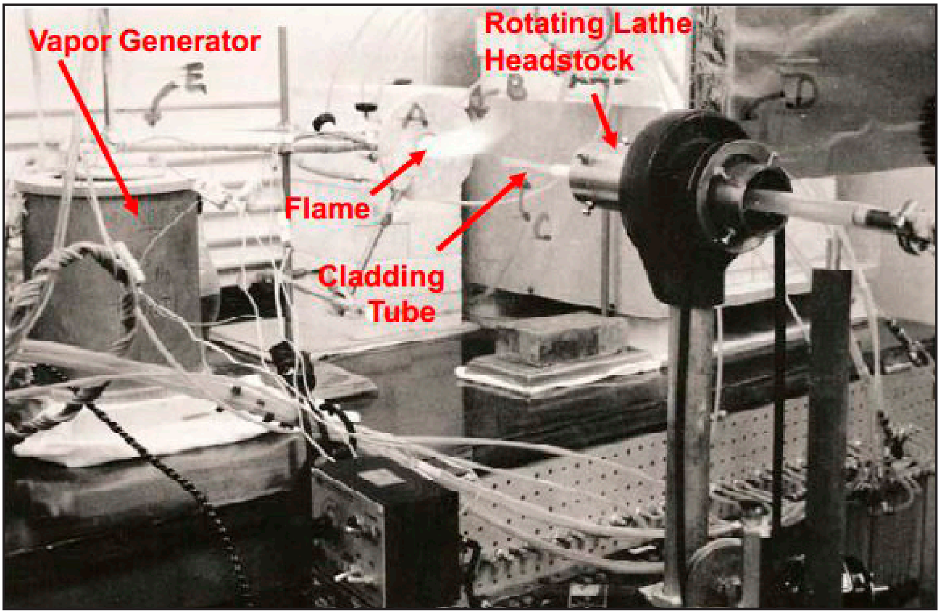

Photo 5. *Fiber Preform Deposition Apparatus 1970 used by Dr. Peter Schultz to make the first low loss optical fiber*

In conclusion, Dr. Hyde's career teaches us that great science does not immediately translate into a commercial success, and that through optimism and vision, new possibilities can be realized. With that, I will leave you with a quote from another great Corning scientist, Dr. S. Don Stookey. [8]

"And as everyone knows, an embryo invention is a fragile flower, easily killed by the pessimism that seems to be a predominant characteristic of anyone over six years old. The pioneering researcher must be an optimist, and strong in character, to overcome the disbelief of his fellows in anything new."

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Understanding Bernard Gitton's Water Clock

by

Benjamin Revis*

ABSTRACT

Bernard Gitton's water clock: fascinating, mesmerizing, simple, complex. These are a few words to describe this beautiful work of scientific art, but how does it work? The following is an effort to simply explain how Gitton's clock works through a logical assembly of the clock diagram and explaining each piece's function.

The purpose of this paper is to share my understanding of the function of Bernard Gitton's famous water clock. For me personally, it was a process that took building a clock to fully appreciate the form and function that is embodied in this beautiful piece of scientific art. I will do my best to simply and concisely describe the water clock's function in the following paper.

Photo 1

Bernard Gitton opens his article "Time, like an ever flowing stream..." in the *Horological Journal*, June 1989 by stating: "We say in French: *le temps s'écoule* - 'Time flows.'" He goes on to conclude "... what is the point of inventing a clepsydra in the twentieth century? … The answer is not a very technical one: this is more a clock for poets than for businessmen!"[1] How true this last statement is. The opportunity to ponder and muse the inner workings while water seems to be the only moving piece of the puzzle is truly a poet or philosopher's effort. This curiosity has gripped me from childhood when I first saw the Indianapolis Children's Museum installation of the Water Clock (Photo 1).[2] Standing 10 meters in height soaring above young and old with a cool blue liquid spilling intentionally, never at rest, fascinating, mesmerizing, evoking curiosity in the simple and the complex.

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[1] Bernard Gitton, "Time, like an ever flowing stresm...," *Horological Journal* (June 1989): 18-20

[2] Image courtesy of The Children's Museum of Indianapolis, used with permission.

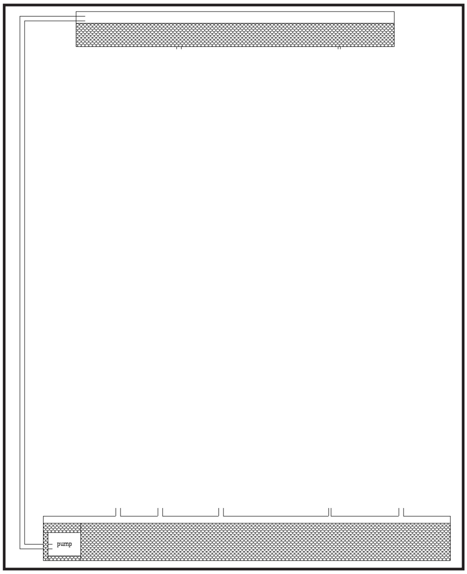

Figure 1

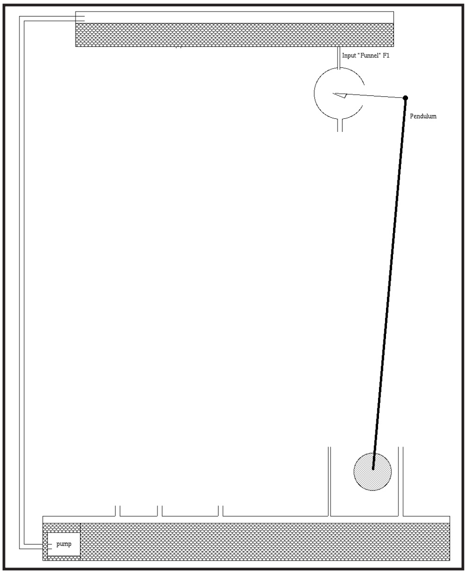

Figure 2

The description that follows will attempt to logically process the function of Gitton's clock. Beginning with the simple, the clock operates by water flowing from a higher potential to a lower. Two holding tanks are needed, one tank at the top providing the potential energy to the system and the one at the bottom to retain the water after it has completed its task (Figure 1). Accompanying these tanks, there needs to be a mechanism adding potential energy to the system. This mechanism could be as simple as someone with a bucket taking water from the lower tank and placing it in the upper; or, more conventionally, a pump. This completes the first block in our assembly, an upper tank, a lower tank and a pump to relocate water from the lower tank to the upper.

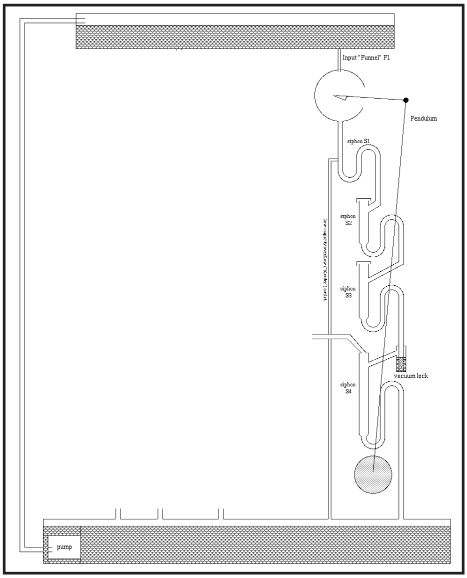

Figure 3

The next block to consider in our logical assembly is the pendulum (Figure 2). The pendulum is sustained by a continuous flow of water from the upper tank into a cup attached to the pendulum arm. This cup empties once every full cycle of the pendulum.

Following the water dollops dispensed from the pendulum cup, we find a series of cascading siphons (Figure 3). These siphons are arranged in such a way that they serve as a frequency divider. Based on the period of the pendulum, the number and volume of each siphon can be determined such that the siphon chain empties once every two minutes. Once this series of siphons empties, it drains into the lower tank.

Next in our block assembly are the two

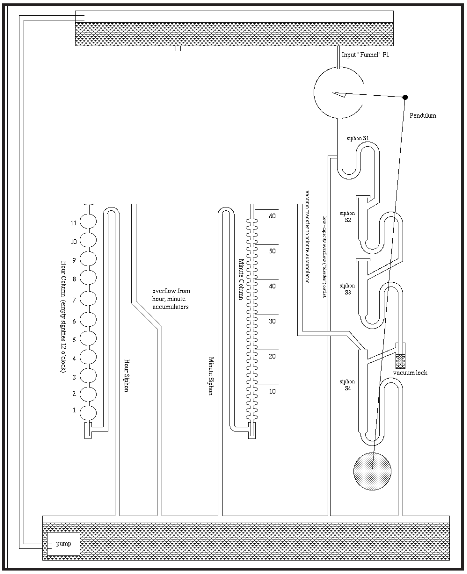

Figure 4

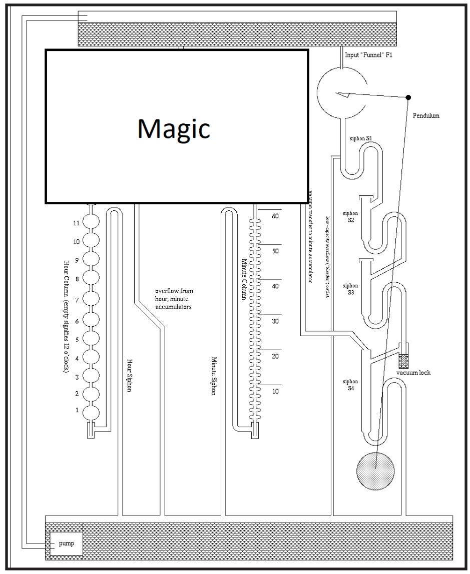

Figure 5

time siphons (Figure 4). These siphons are straightforward in their function. When they are full, they will cycle. The challenge, and a key part of understanding what will be discussed next, is that for each of the time siphons, they require a specific volume of water to be added at specified intervals (two minutes and one hour respectively). The volume of each interval is nondescript only requiring that it is consistent between intervals; the consistent intervals represent the incremental passage of time.

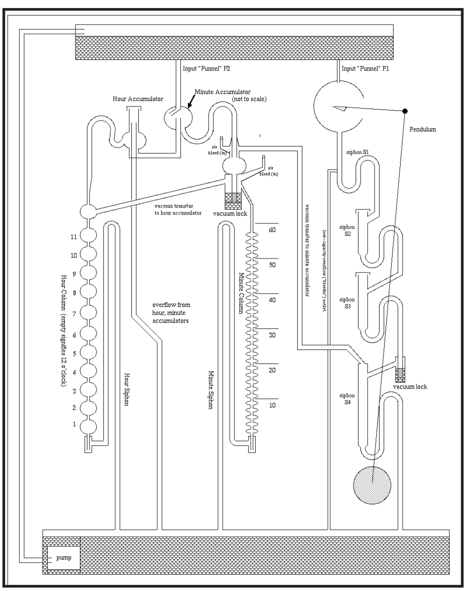

Figure 6

The next block to consider is where I believe magic happens (Figure 5). For many just believing the magic happens allows for all to be right in their world. Yet, for the rest of us who want to really understand what is going on, we desire to know how the interplay of the three main siphon systems interact to tell each appropriate volume to be dispensed at the correct time. Jumping back to the concept of magic, those who say 'magic happens' are not too far from the truth. Magic usually entails a bit of sleight of hand or misdirection. This clock design ingeniously employs a similar misdirection (Figure 6).[3] In looking at the cascading siphons, the last siphon is the only one not open to atmosphere but it is connected via an air column up to a special trap that is connected to the awaiting

[3] Base image from David M. MacMillan website "Delightful Machines." Accessed 8/20/2019. <http://www.marcdatabase.com/~lemur/dm-gitton.html>

minutes' volume. We will look more at how the minutes, hours and decision trap are connected and interact shortly. For now, as the frequency divider completely drains down, our eyes follow the falling liquid. What we do not see is the effect on the transparent liquid (air) in the connected column. The air is brought to a more negative pressure drawing the awaiting minutes' volume over the top of the siphon bend and draining into the minutes' column. This is the magic; blink and you miss it.

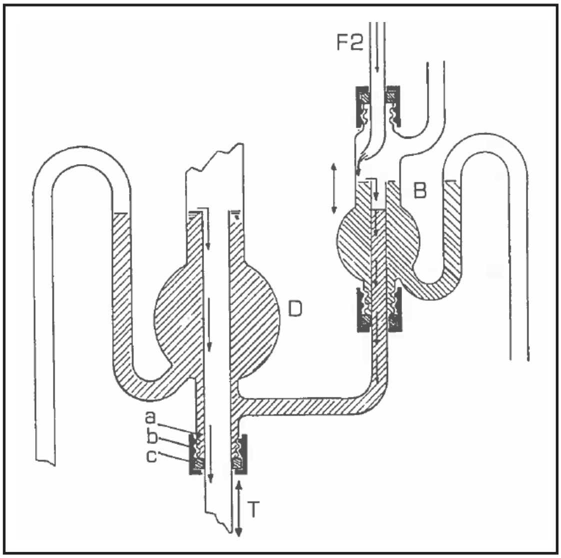

Figure 7

Now that I have let the magic smoke out let us look at how the minute and hour volumes are filled, regulated and triggered (Figure 7).[4] In order to fill the minutes, a continuous fill valve is set to dispense water along the side of the volume until the water level overflows into a drain. The drain height is adjustable to a certain extent allowing for fine tuning of the minutes' volume. The water that drains out of the minutes' volume through the drain begins to fill the hours' volume from the bottom up. Again, an adjustable drain is utilized to make minor adjustments to the hours' volume. The water that spills over the hours' drain just returns to the bottom holding tank. There is a fine balance that needs to happen with total volume of liquid in the system and the rate at which the continuous fill valve is set: too much flow, you use up a lot of potential water from the upper holding tank unnecessarily; too little, the hours' volume may not be completely full before being called upon for its purpose.

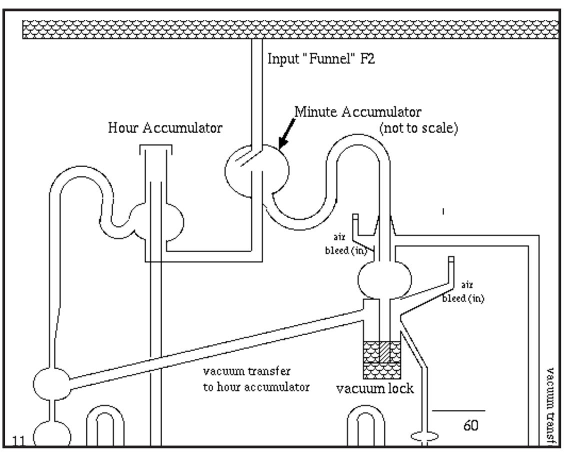

Figure 8

The last piece that needs to be looked at is the decision trap (Figure 8).[5] This piece is beautifully thought out: when the frequency divider siphon empties, the only air space impacted directly affects the minutes' volume only (top part of the trap). And when the minutes' column siphons, the air space impacts only the awaiting hours' volume (bottom part of the trap), though the minutes' and hours' volumes should both be called upon within seconds of each other.

[4] Image from *Horological Journal* (June 1989): 19.

[5] Base image from David M. MacMillan website "Delightful Machines." Accessed 8/20/2019. < http://www.marcdatabase.com/~lemur/dm-gitton.html>

This effort has taken me on a wonderful journey from childhood fascination to realizing a far better hands-on understanding for this beautiful piece of science and art. From this process, my colleague and I have communicated with Bernard Gitton and his wife. We have also contacted the company (V.E.R.A.L. Glassblowing Company, France) that currently possesses the blueprints to many if not all the Gitton clock designs. It may be interesting to know that the clock designs did change over time to reflect Gitton's desire to make his time piece more accurate. In reference to accuracy, this clock will never inherently be a true time piece by horological standards, but more specifically as Gitton stated "... this is more a clock for poets than for businessmen!"

In conclusion, our efforts have taken this clock along with other scientific glassware, geological findings and physics phenomena on stage with musical accompaniment as a public performance to the Iowa City community.

ACKNOWLEDGEMENTS

This project would not have been possible without funding from the University of Iowa Creative Matches Grant – Office of Vice President of Research, Bernard Gitton and his wife Francine for their willingness to answer questions, and Jean-Francois Charles – Composition assistant professor School of Music, The University of Iowa. I would like to thank The University of Iowa Department of Chemistry for their continued support.

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Coyne, Mara
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Cross, Zachary
Daenzer, David
Daenzer, Katrina
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Demoisson, Frederic
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Ditchburn, Brian
Dougherty, Mary
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Dural, Nezih
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Evans, Micah
Ferguson, Sabrina
Ford, Kiva
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Foster, Scott
Fowler, Suellen
Fox, David
Friedman, Charles
Furukawa, "Friko" Yuji
Galbreath, Nolan
Garon, Nathalie
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Gillen, Jason
Ginart, Cedric
Goldschmidt, Eric
Greenfield, Joshua
Gregar, Joseph
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Greico, Annette
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Guerra, Corina
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Hatz, Doni
Hayman, Howard
Henthorne, Timothy
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Hitchcock, Stephanie
Hodgson, Carolyn
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Hoffman, Daniel
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King, Eamon
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Kohl, Jesse
Koob, Dr. Stephen
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Kornbluh, Marc
Kotera, Masashi
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Legge, Marie
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Legge, Sherri
Lehberger, Ken
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Lore, Melissa
Lore, William
Losch, Sr, Ronald
Lynch, Colin
Magfoth, Nancy
Manezzo, Bernadette Sarah
Martin, Dave
Mask, Harrison
Mason, Mike
Mathews, Victor
Matrone, Liberata
Mattei, Andrew "Drew"
McClelland, Jeremiah
McCrossan, Nolan
Meyer, Kyle
Miller, Chris
Milutinovich, Nicholas
Milutinovich, Zorko
Mobsby, Sarah
Moncke, Ph.D., Doris
Moraine, Erich
Morehart, Casey
Morehart, Tiffany
Mori, Laurent
Moss, Peter
Moss, Roxanne
Mukhtar, Sabeen
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Nagami, Craig
Nakamura, Tsuyoshi
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O'Donnell, Dylan
Owoc, Brian
Paris, Klaus
Pearson, Ian
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Pickett, Annalee
Pittner, Marianna
Plati, Marinella
Ploucher, Richard
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Ponton, Robert
Prasch, Sally
Preston, Mitch
Quack, Jürgen
Ramirez, Arturo "Art"
Ramon Herrera, Joaquin
Ramos, Pamela
Revis, Arwen
Revis, Benjamin
Revis, Emma
Revis, Logan
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Rosenberry, Zack
Rossi, Christina
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Schiaffino, Michael
Schnuckel, David
Schwandt, Brian
Sekiya, Koki
Severance, Kathleen "Katie"
Shanafelter, Allie
Sinclair, J. Mark
Singer, Robert
Sliwoski, Laurie
Sliwoski, Phillip
Smith, Daryl
Smith, Jordan
Smith, Lorraine
Smith, Preston
Smith, Rick
Smith, Todd
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Suerfu, Dr. Burkhant
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Taku, Terada
Tatsushi, Iguchi
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Teaford, Rhonda
Thackery, Ross
Thorn, Brent
Tossmann, Tifa
Trankina, Joseph
Trembly, John "Mike"
Turner, Brad
Uesato, Lianne
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Vo, Minh
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Wargo, Andy
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Wentzel, William
West, Jackie
West, Joseph
Wingard, Brad
Wu, Joanna
Zuber, Sierra
Zuhlke, Christopher